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Simple Method for Emulsifying Oils

By CHARLES F. GOLDTHWAIT

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During a number of years' work on textiles the writer has had occasion a number of times to prepare emulsions of ordinary oils. It was found that, in general, the literature on the subject did not prove to be much of a help. As a result of a great many experiments, both successful and unsuccessful, a comparatively simple method has been found which works very well in most cases where a soap emulsion of an oil will answer the purpose.

In a number of the trials better emulsions were made by soap and simple hand stirring than by special emulsifying agents or high speed mixers.

Naturally a little experience with this method is necessary for the best results. Each oil, and each soap that is to be used as an emulsifier is something of a special problem. There are evidently no hard and fast rules that can be followed.

THIS method hinges on many observations that one of the real secrets of making such emulsions is to have present in the emulsifier just the right amount of water. It appears that under the most favorable conditions the water, soap and oil build up some definite sort of structure which can be diluted to a stable emulsion.

As is usual in colloidal and textile problems it is none too certain just how many of the details and precautions are necessary. However, the process does not seem overburdened.

Briefly, to prepare an emulsion from a given oil using a given soap as emulsifier start with a strong solution of the soap, such as a concentration of 40 per cent of the fatty acids. If the soap has to be prepared it can be made at this concentration, or if already available a solution of this strength can be made. Then take in a beaker an amount of the strong soap solution equivalent, when calculated as fatty acids, to 5 per cent of the weight of oil to be emulsified, and add a little of the oil. Stir the concentrated soap solution and the oil together using a flat stick. They should soon combine to a thick paste. If they fail to do this, feed in a very little water, then a little oil, and so on slowly until, when the right amount of water is present, the mass will show a characteristic thick consistency. It is hardly possible to describe just what takes place but it is usually obvious when the mass becomes properly emulsified that some definite change has occurred. The mixture ceases to look oily.

As an example, an emulsion in the proper form at this concentrated stage may stand up as an almost solid mass; it will vibrate like a jelly when hit with the stirring stick; and may even transmit the vibration through the beaker to the hand in which it rests.

When this stage is reached the rest of the oil should be added, preferably a little at a time, but no more water until all of the oil has been worked in. When adding the rest of the oil the emulsion may appear to be broken down but it should return to the quivering jelly form after each oil addition.

After completing the concentrated emulsion, dilution should be started by stirring in a very little water. At first a thick paste will be obtained, which will thin out with successive additions and then go over to a thin milky emulsion. The results will not be stringy, pasty mixes, but thin liquids even at such concentrations as 50 per cent oil.

This method was worked out several years ago but there was recently occasion to use it again, so a few emulsions were made as a recheck on the principles involved. A brief description of specific mixtures and results will probably be more helpful than a more detailed general explanation.

When working out a new emulsion, it is better to start with concentrated soap and some of the oil, then introduce small amounts of water and perhaps more oil until the mass emulsifies to the right consistency. After the amount of water has been determined it may more conveniently be present in the soap at the start for duplicate future emulsions.

Examples of Emulsions Made

The first emulsion to be described was made with an olive oil never tried previously but a good emulsion was made the first time. It was started with 6.25 parts of

40 per cent solution of potassium and ammonium oleates mixed in a one to one ratio, all on fatty-acid basis. Having weighed out 50 parts of ordinary denatured olive oil, about a fifth of it was stirred into the soap.

The oil-soap mixture was really oil with finely divided soap suspended in it, pasty but not a smooth paste. Water was slowly worked into it at this stage and after about 5 parts were added the soap became sufficiently "hydrated" so that a change could be seen, and then the emulsion smoothed out and thickened to the jelly stage already described. It took more oil in several additions up to perhaps three-fourths the total amount, when it refused to come back to the desired form but became thin and oily with the "hydrated" soap suspended in it. After a rather large amount of stirring and the addition of concentrated ammonia (to avoid using water at the same time) slowly up to a persistent odor showing free ammonia in the mass, the emulsion came back to the desired form. It was then diluted by adding very small amounts of water, and then larger amounts as it became thinner, to 300 parts.

This emulsion was creamed but very satisfactory, and no free oil had separated within the first two days.

The emulsion should not break part way through the mixing and this was attributed to one or both of two things, the addition of water a little late in the process, and the presence of too much free fatty acid.

On a second mixing of the same ingredients the 5 parts of water were put in as soon as enough oil was mixed with soap to prevent frothing; and the oil was made definitely alkaline with ammonia to neutralize the free fatty acids. The resulting ammonia soap could be seen in suspension. This mix went through quickly, forming the characteristic jelly-like paste at once and reforming it quickly after each addition of oil.

This is obviously the correct way to make this particular emulsion. However, a third mixing was made as a check on the influence of free fatty acids. The five parts of water were put in at the start but the oil was not neutralized. This mix went through properly up to the last addition of oil, when it slowly broke down to a thin oily paste with the "hydrated" soap present as a second phase. The gradual addition of ammonia and long continued stirring brought this back and it was diluted in the usual manner to a thin milky stable emulsion.

The early addition of water made some improvement but the neutralizing of the free acid was also necessary for a good result.

This may involve a question of pH as, apparently, emulsions made with soap are apt to come out better if alkaline in reaction. A pH of 8.2 has been recommended, with 8.5 as a maximum (Travis).

All three emulsions were about the same in stability and neither of them showed any free oil in two days.

Another recent successful emulsion was made from castor oil, 75 parts with 16.7 parts of a 1:1 mixture of 30 per cent potassium and ammonium oleates, equivalent to 5 parts of oleic acid. It is probable that the 11.7 parts of water present at the start of the mixing was not quite enough for the best result but the emulsion answered its purpose. It creamed without showing a very definite cream line and liberated only a small amount of free oil even after several days.

As a third example an emulsion using a more extreme type of fat was made with hydrogenated cocoanut oil which was used melted but not very hot. It will illustrate the method and show even more striking the hard vibrating jelly stage. It might be pointed out here that it is well to have the soap mixture somewhat alkaline with ammonia. Then in the case of an oil like cocoanut which is usually low in free fatty acids it may not be necessary to do any neutralizing.

Starting with 25 parts of a mixture of potassium and ammonium oleates in the proportion of one to one and of a total fatty acid concentration of 20%, 75 parts of the melted oil were gradually stirred-in. This mixed readily to a hard vibrating mass which transmitted the vibration to the hand. After slowly diluting, the emulsion was a thin milk. It creamed somewhat but was very stable after a number of days. There was no evidence of free oil. In this case, however, any free oil liberated would be present as a white solid and would hardly be noticed unless considerable in amount.

Miscellaneous Details

It is believed from special tests such as the one already described that free fatty acids may interfere with the proper emulsification of an oil. In one case an old rancid cocoanut oil made a good emulsion after the free fatty acid was neutralized by using a slight excess of ammonia. If the free fatty acid content is high, and it is neutralized, this amounts to using more soap and less oil, and a correction may be necessary.

It is probable that a certain amount of free fatty acid can be tolerated, and there has been some evidence that stearic acid itself can be emulsified by a modified method. Again, it is clear that each case needs special attention.

As far as known the water-soap ratio is not necessarily constant if the amount of soap is varied for a given amount of oil.

Frothing of the soap before emulsification or during the process seems to interfere and to produce an unsatisfactory result. High speed mixers sometimes cause such frothing too early in the process.

A thin flat stick makes a much more effective mixer than a glass stirring rod and has been used in much of this work.

These emulsions may cream out in a short time or overnight but are readily stirred back. They usually show very little if any free oil for a long time if kept in closed containers.

Since the emulsions are thin and milky by the time they are diluted to 50 per cent oil it is possible to make very fluid mixtures of high concentration.

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Shrinkage of Cotton and Linen Woven Materials--II

By G. H. JOHNSON

(Continued from page 584, Sept. 25th issue.)

(*g*) *The Greatest Shrinkage Occurs During the First Washing*

As a general rule, it is the shrinkage that occurs during the first washing that is the most serious. It undoubtedly is true that a slight additional shrinkage may occur through the third washing. However, the additional shrinkage is so slight after the first two or three launderings as to be insignificant. It, of course, is true that instances have been reported wherein consumers claim no shrinkage at all after the first washing, but a serious contraction suddenly after the tenth or twelfth trip to a laundry. As will be seen later, variations in pressing technique may be responsible for such instances. In other instances the customer has been misled in his or her conclusions. There is no *washing reason* why a cotton or linen woven fabric can be washed ten times without shrinking and then suddenly shrink during the next laundering. Other factors—including misinformation—are responsible for such instances and not the washing process. In the experience of the L. N. A. Department of Research, shrinkage, except that occurring because of variations in tension applied at the time of pressing, is practically complete at the end of the third laundering. The vast bulk of the total shrinkage obtained takes place during the first laundering, as is shown in Table XI.

This table tabulates the shrinkage obtained during one and two launderings, one set of measured samples having been laundered with woolen lots at 80° F. and the other at 120° F., with a cotton classification. The samples were tumbler dried after each washing and measured dry.

Table XI

		Shrinkage in Inches Per Yard			
	Number of	Woolen formula at 80° F.		Cotton formula at 120° F.	
Fabric	Launderings	Warp	Filling	Warp	Filling
A	1	4.6″	1.7″	5.4″	1.6″
	2	5.3	1.7	5.7	1.8
B	1	3.0	0.5	3.5	0.4
	2	4.1	0.5	4.3	0.6
C	1	2.0	1.5	2.0	1.8
	2	2.2	1.5	2.2	2.0
D	1	3.3	1.6	3.0	1.3
	2	3.8	1.7	3.6	1.5

A study of Table XI, which is typical, reveals the fact that the second laundering increased the warp shrinkage from 0.2 of an inch to 1.1 inches per yard, in the case of the materials in the preceding table. Calculated in terms of percentage, the amount of warp shrinkage occurring during the first laundering compared with that obtained after two washings varied between 73.1 and 94.7 per cent; the amount of filling shrinkage obtained after the first washing varied between 86.6 and 100 per cent of that secured at the end of two. With repeated washings, slight added contractions may occur, due chiefly to pressing, but it is plainly apparent that by far the bulk of the total shrinkage obtained takes place during the first washing.

(*h*) *The Speed of Shrinkage*

Another very interesting fact concerning the phenomenon of shrinkage has to do with the speed or rapidity with which the contraction occurs. All that seems to be necessary is complete saturation and even a partial saturation with moisture has been found to cause some shrinkage.

The rapidity with which contraction occurs probably is influenced somewhat by the presence of sizing, as previously discussed, which is of interest theoretically. However, during an actual washing, the great bulk, and frequently all, of the sizing is removed, thereby permitting normal shrinkage to occur in any event.

In Table XII are shown the results of a test conducted with a white cotton shirt. These data illustrate how rapidly shrinkage occurs with lightly sized and unsized fabrics. After being measured, the shirt was soaked for 10 minutes in cold water at 65° F. It was measured again and then allowed to soak an additional 50 minutes, making a total immersion of one hour. The garment was again measured and allowed to air-dry, after which it was remeasured. It should be noted that practically as much shrinkage was secured after a 10-minute immersion as during one hour of soaking in cold water.

Table XII

	Original	Soaked for 10 minutes	Soaked for one hour	Air-dried
Collar	14⅛″	13½″	13½″	13½″
Sleeve	20	19½	19	19
Cuff	2¾	2⅝	2⅝	2⅝
Button yoke	30	29⅜	28⅞	28⅞
Back	32	30⅞	30⅞	30½

A similar fact was observed when conducting tests with an overall sample. A piece of cloth 48 inches long and 8 inches wide was immersed in water for 45 seconds during which time a 2¼-inch warp shrinkage occurred. The following measurements were recorded:

Original Length	Soaked in cold water for 45 seconds	Soaked in cold water for 30 minutes
48″	45¾″	43.1″

A third series of tests also established the fact that shrinkage, except for heavily sized fabrics, occurs very rapidly. In these tests five measured samples of overall materials were immersed in a neutral soap solution at 130° F. for 2 minutes, after which they were measured. They were then immersed for a total of 10 minutes and again measured. The following warp shrinkages were obtained:

Table XIII

Warp Shrinkage in Inches Per Yard of Material

After soaking 2 minutes	After soaking 10 minutes
2.5″	3.0″
2.7	3.2
2.7	3.6
2.5	3.3
2.8	3.3

In summary, once cotton and linen materials become thoroughly saturated with moisture and the sizing is completely penetrated or removed, the fabrics shrink, the amount of contraction depending upon the degree to which each fabric was stretched during mill processing. All that a preshrinking process is designed to do is to remove any shrinkage due to stretching prior to cutting and tailoring.

(*i*) *Comparative Warp and Filling Shrinkages*

Examination of any of the preceding and following tables establishes the fact that the warp shrinkage frequently is greater than that of the filling. Although this general observation is not always true, it has been learned that by far the majority of the cotton goods tested developed a greater warp than filling shrinkage. This fact further indicates that the constant pulling action undergone by warp threads during manufacturing has much to do with the problem of shrinkage. During carding and spinning, for instance, the fibers are constantly being pulled or stretched. During beaming, warping, and weaving the warp again is in a stretched or tautened condition. After kier-boiling, as previously shown, tension again is applied length or warp-wise.

Table XIV is of interest since it shows the average shrinkage obtained with 127 samples of sheeting during actual laundering[1]. It again brings out the fact that in by far the majority of cases, unbleached sheetings apparently being an exception to the rule, the warp shrinks more than the filling. The Cotton Textile Institute reports that 45 sheets representing several brands developed an average shrinkage of over 4½ per cent during laundering.

[1] Journal of Home Economics, p. 429, June, 1928.

Table XIV

Sheeting Shrinkage

Kind of Sheeting	Weight	No. of sheets tested	Shrinkage in inches per yard		Shrinkage in inches based upon a 103″ length	
			Warp	Filling	Warp	Filling
Unbleached	Light	3	2.7″	3.2″	7.7″	9.1″
Unbleached	Medium	3	2.1	3.0	6.0	8.6
Unbleached	Heavy	4	2.2	2.6	6.3	7.4
Bleached	Light	19	1.9	0.46	5.4	1.3
Bleached	Medium	50	2.2	0.43	6.3	1.2
Bleached	Heavy	48	1.8	0.6	5.1	1.7

The figures in the last two columns are based upon a 103-inch hemmed length because the Cotton Textile Institute has reported that 208 sheets representing 23 mill brands had an average length of 103 inches after hemming even though the torn length was 108 inches.

Tables XV and XVI further illustrate the fact that the warp shrinkage usually is in excess of the filling contraction. They, furthermore, offer ample proof that there is need for using a satisfactory preshrinking process in the manufacture of any material that is to be tailored to fit. A 3-inch shrinkage in a sheet, as previously mentioned, may not prove especially serious, but a 3-inch shrinkage per yard in the case of a slip cover or a shirting causes consumer dissatisfaction.

Table XV

Furniture Cover Shrinkage

	Shrinkage in inches per yard, tap water		Shrinkage in inches per yard at 120° F.	
	Warp	Filling	Warp	Filling
1	2.8″	....	3.4″	0.6″
2	3.4	1.3″	3.6	1.3
3	3.5	1.8	3.4	1.9
4	3.4	1.3	3.5	1.9
5	2.3	1.1	2.8	1.1
6	3.0	0.5	3.0	0.5

Table XVI

Washsuit Shrinkage

	Shrinkage in inches per yard	
	Warp	Filling
Unsponged, 1	0.4″	0.2″
Unsponged, 2	2.9	0.9
Unsponged, 3	3.7	1.0
Unsponged, 4	1.6	0.5
Unsponged, 5	2.5	...
Sponged, 1	...	...
Sponged, 2	0.9	...
Sponged, 3	1.4	...
Sponged, 4	0.9	...
Sponged, 5	1.1	...

In Table XVI it will be noted that in addition to the warp shrinkage being greater than the filling contraction, the unsponged washsuit material shrank to a greater degree than did the corresponding sponged or partially preshrunk samples. These data indicate, however, that sponging alone does not entirely preshrink washsuit fabrics.

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PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

OCTOBER 9, 1933

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Joseph F. X. Harold, Chemist, 50 East 41st St., New York, N. Y.

* For re-election.

We beg to call your attention to the following excerpt from Article VIII of the Constitution:—

"Counter nominations of any office, signed by at least ten members, may be filed with the Secretary not later than November 1."

For the Nominating Committee.

A. Newton Graves,
Secretary

THIRTEENTH ANNUAL CONVENTION

FRIDAY EVENING SESSION

September 8, 1933

THE Thirteenth Annual Convention of the American Association of Textile Chemists and Colorists convened at nine-fifteen o'clock in the Florentine Room, Congress Hotel, Chicago, Illinois, William H. Cady, Vice-president of the Association, presiding.

Chairman Cady: Gentlemen, once again I have the pleasure as well as the duty of welcoming you to the Annual Convention of this Association.

As some of you already know, our esteemed President, Dr. Rose, is unable to be with us this time. He has a bad attack of hay fever and his doctor has forbidden him to come to Chicago. In fact he is now in the Southwest in the mountains recuperating. He is extremely sorry to be away and has asked me to express his great regret.

The first speaker on this evening's program is a man whom you all know, I think, much better than you do me, Dr. E. H. Harvey, Director of the Bureau of Standards of Montgomery Ward & Co. He is going to give an illustrated lecture on the subject "Chemistry in Merchandising." Dr. Harvey!

Proceedings of the American Association of Textile Chemists and Colorists

Chemistry in Merchandising*

By **ELLERY H. HARVEY**
Montgomery Ward & Co.

AS NEARLY as the record discloses, chemical testing of foods and drugs began at Montgomery Ward & Co. shortly after the passing of the Foods and Drugs Act of 1906, mention being made of it some time later in Catalog 82, issued in 1912. The demand from other departments for a similar service grew until in 1916 a staff big enough to cope with additional problems was provided. While young in years the founding of the Laboratory marks an important step in the evolution of merchandising and even at such a relatively late date represented a departure in business methods that was unique. As old as Chemistry and Physics were as abstract sciences, and even as handmaidens to certain fundamental industries, this is the first application of science to the solution of the multitudinous problems that arise in merchandising some 40,000 articles by retail.

In 1872 when A. Montgomery Ward originated the idea of buying by mail, the frontier had not closed. Roads were poor and transportation expensive and slow. Towns were scattered. Those living in rural communities were barred from wide selection, up-to-the-minute styles and low prices due to this isolation. For those living in rural communities, barred from contacting markets, Mr. Ward conceived the idea of carrying the market to the consumer which was done through the medium of the printed word. This unique type of merchandising, logical as to plan and successful immediately in execution, was accompanied by problems peculiar to itself. There was need for establishing confidence in the customer's mind that he would get merchandise exactly as described and this was done by the use of the guarantee "Satisfaction or Your Money Back."

The Catalog being the only salesman and a silent one at that, had to carry a wide selection of fresh merchandise in the many wanted colors, price ranges, and sizes through the use of attractive layouts accompanied by accurate and informative descriptions. There had to be prompt shipments and rapid turnover and, at the same time, low inventories yet avoid possible omissions. Keeping pace with changes of style and price made a flexible organization necessary and this resulted in the establishing of nine strategically located mail order houses.

Real contributions to the solution of many of these problems has been made by the technical department now known as the Bureau of Standards. The part played by this group can best be illustrated by showing how our work affects the various activities of the business.

Organization

The work of Ward's Bureau of Standards consists of three major activities: analytical, shopping, and garment sizing. This arrangement makes possible a well rounded and balanced service that begins with the buyer and his problem of making wise selections, carries through with an analysis of competitive merchandise, catalog presentation, and sales promotional plans, and ends with a scrutiny of customer service.

The Analytical is divided broadly into three units: administrative, chemical analysis, and physical testing. No restrictions stand in the way of its use and no argument is needed to justify its existence. The cost of operation is considered a general expense to further encourage the use of its facilities.

The mechanics of getting a sample analyzed consists in sending it to the Laboratory, accompanied by a Request for Analysis form in duplicate, indicating specifically what is desired. If something unusual is wanted the person originating the request usually discusses his problem with the laboratory director, and in most cases leaves the entire matter in our hands. The requests are dated and numbered as received, and each problem assigned to the staff member best qualified to handle it. This is done in order of receipt although exceptions are frequently necessary to meet catalog schedules or to make a train to a distant point. The duplicate copy of every report is filed by year and subject matter and the original copy goes to the person originating the request. These records now constitute an excellent background for comparative purposes.

The chemical and physical testing staffs supplement each other, few requests for assistance being confined strictly to either one alone. Standard methods of analysis are employed whenever possible but many, particularly in physical testing, must be developed and a machine

*Presented at Friday evening session, Annual Convention, Chicago, September 8.

shop is maintained for designing and building needed equipment. A good working library provides literature facilities in the field of fundamental and applied science.

One room is set aside for textile analysis fully equipped to handle all requests for work in this field. The usual routine tests involve microscopic identification of fibers and type of weave, weight per square yard, thread count, tensile strength, tubfastness, sunfastness, shrinkage and quantitative estimation of fibers. In addition, where appropriate, tests are made for waterproofness, absorbency, metallic weighting, abrasion resistance, mothproofness, mildew resistance, twist of yarns, degree of mercerization, identification of dyes, flexing tests, per cent of sizing, hosiery analysis, customers' complaints. What we call textile analysis involves such a variety of materials as underwear, hosiery, dresses, coats, suits, millinery, blankets, towels, typewriter ribbons, stationery, wall paper, floor coverings, drapes, window shades, notions, tents, oilcloth, upholstering.

In the Chemical Laboratory facilities are available for the analysis of items in the hardware division for hardness, carbon content, constituents, metallography, amount and quality of plating, tensile strength and salt spray corrosion.

The examination of paints, varnishes, and lacquers includes chemical analysis, weight per gallon, hiding power, viscosity, abrasion resistance, brushing qualities, drying time, artificial and roof weathering.

Foods and drugs occupy much of our attention. The organic chemists responsible for this work also include shoe polishes, floor waxes, inks, washing and cleaning compounds, bactericidal value of antiseptics, adhesives, fertilizers, insecticides.

The analysis of water, coal, lubricating oil, gas, roofing and similar items are analyzed both for our own information in connection with supplies and for re-sale.

Separated from the Chemical Laboratory to minimize corrosion, noise and vibration, the physical testing unit includes radio and electrical measurements, a fireproof room for fire hazardous merchandise, a constant temperature room for refrigerator testing, a machine shop and a dark room for photomicrography, spectroscopy and X-ray analysis, with miscellaneous equipment for tensile strength tests on rope, wire, leather and compression tests on furniture, etc. Auto radiator efficiency measurements, mattress tester, chinaware impact, felt base rug abrasion machine, indentation apparatus, foot-candle meter, pyrometric equipment, etc.

Here cream separators are checked for skimming efficiency, incubators for ability to hatch large percentages of fertile eggs, gasoline engines and electric motors for horse power ratings, dirt picking capacity of vacuum cleaners, efficiency of washing machines, sewing machines and dozens of household items tested for their satisfactory performance in the home.

Aid to the Buyer

The Buyer in our organization is the man who contacts the markets for merchandise in his line, selecting for resale those items for which a demand exists, taking into consideration quality, price, distribution and delivery. In essence, the Buyer is a full fledged merchant, specializing in certain lines, such as sporting goods, luggage or paint, in which he must give due regard to catalog arrangement, publicity expense and inventory control. The Buying organization of a mail order house is in essence therefore an aggregation of merchants, each one an expert in his field.

As the Buyer contacts his sources of supply, he is under the same barrage of high pressure selling even as you and I when we function as ultimate consumers. Facts developed by the Laboratory act as a common denominator in reducing all this sales talk to something definite and tangible. Capable as any Buyer may be most of the points of major importance from a quality standpoint cannot be determined by mere inspection. A carefully considered report from unbiased laboratory experts who interpret their data in the light of consumer needs, special guarantees and competition makes it possible for the buyer to make his contacts with confidence, and in addition releases a tremendous amount of time for other work. In effect, the Laboratory staff are merchandise counselors to the Buying Organization.

In any such program obviously the matter of specifications is given great consideration. It is the very essence of the mass production, mass distribution, mass consumption idea.

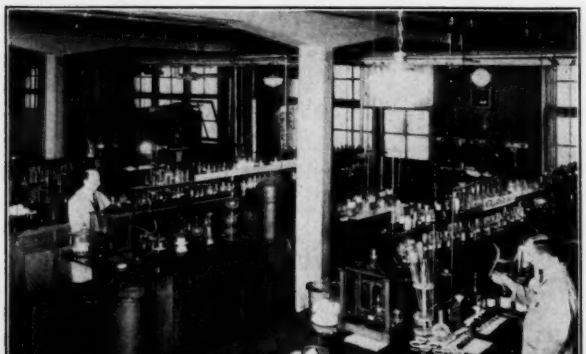
Aid to the Operating Department

Only part of the job is done after merchandise is analyzed, selected and bought. Each incoming shipment must be checked to determine whether purchasing specifications have been satisfactorily met. Items rejected by the Laboratory are returned to the manufacturers for credit. Obviously there is but little comfort in such a picture for the manufacturer who knowingly or unwittingly permits a substantial variation of quality or quantity to occur in his product.

Incidental to the operation of a business involving nine mail order plants and five hundred retail stores there are innumerable problems, many of which are referred to the laboratory. "Maximum service with minimum expense" is the keynote which makes it possible for us to aid the Purchasing Department buy better coal, fuel oil, paper, twine, ink, adhesives, soaps, disinfectants, paint, varnish, floor oils and waxes, shipping cartons, etc.

Complaints of a technical nature are referred to us. This makes possible direct customer contact which is mutually helpful; the inquirer receives a prompt and intelligent reply on the one hand, and we learn some of the things that bother the ultimate consumer.

Below—A corner of the chemical laboratory of Ward's Bureau of Standards.

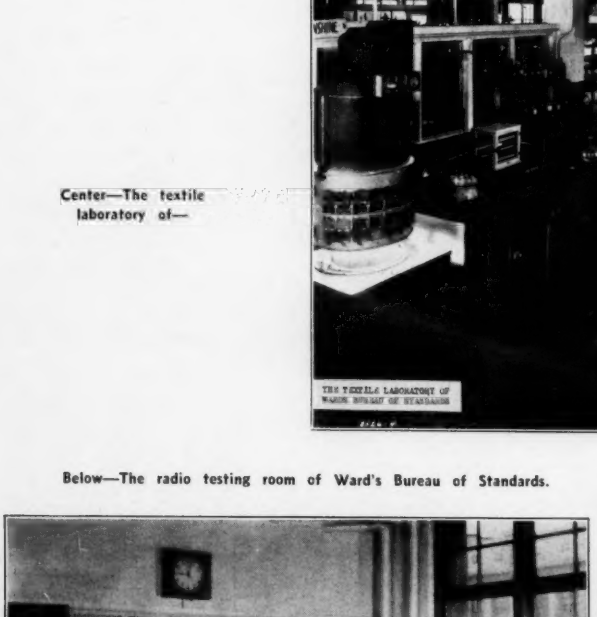


Above—The shopping unit of Ward's Bureau of Standards.

Center—The textile laboratory of—

—Ward's Bureau of Standards.

Below—The radio testing room of Ward's Bureau of Standards.



Above—Part of the physical testing room of Ward's Bureau of Standards.

Then there are many who write direct for help in connection with some problem of the home or farm. Among recent inquiries were included requests for the hardness of water, economic value of a mineral that was found locally, tannage of small skins, value of ultra-violet lamps, type of fertilizer for onions, information on weighted silks, determination of wool in a fabric.

The preparation of instructional literature occupies a part of our time. We are anxious that the merchandise perform satisfactorily and economically hence care is taken to indicate proper conditions of use. This involves unpacking, assembling, and actual operation. This activity brings weaknesses to our attention if there are any and helps to eliminate returns.

The proper sizing of garments is much more necessary in merchandising by mail than in over-the-counter selling because in the former the try-on feature is not available except after delivery has been made and, if the garment does not fit it necessitates either a remodeling or a return of the merchandise for a different sized article. Staff members, skilled in the subject of making body measurements and in writing specifications, constantly review our size specifications to keep them up to date with style trends and to make examination service efficient.

Merchandise Comparisons

To know what competition is doing is almost as important as knowing your own objectives. The company sets for itself the goal of selling the best merchandise obtainable at the price at which it is sold, and a constant stream of nationally advertised washing machines, vacuum cleaners, cream separators, gasoline engines, electric motors, incubators, harness, fencing, roofing, paints, just to mention typical examples, pass through the laboratory for a point-to-point comparison with our own. The results of some of this work finds its way into the hands of the Buyer and Manufacturer, so that weak lines may be strengthened; some of it is utilized by the Sales Manager in planning his selling campaign. The Advertising Department leans heavily on such reports in the preparation of copy.

These comparisons include a detailed analysis of retail and mail order selling with respect to assortment, price lines, eye appeal, ease of ordering, layouts, space allotment, feature presentation, exclusive features, collateral advertising and service. In a very real way, therefore, the entire organization is assisted by a scientific approach to every problem of a merchandising nature.

Legal Requirements

The local merchant on intra-state shipments has at most only the legal requirements of his state to watch, and in the larger cities, the activities of the Better Business Bureau. The mail order house on the contrary, must meet the legal requirements of forty-eight states and of the Federal Government as well.

Labels, both on nationally advertised items and those put on the market under our own name, receive careful attention. We pass upon the agreement of label claims and actual composition, compliance with the requirements of the Food and Drug Act, Caustic Poison Law, Postal Regulations and suitability of a particular product for an intended use. Special requirements of certain states with respect to bedding, paints, etc., are carefully followed.

Rules promulgated for the guidance of the advertising department have their basis in the case law and stipulations issued by the Federal Trade Commission. This is the agency set up by Congress to prevent unfair competition in intra-state trade. It does not take jurisdiction over intrastate business and for that reason the national distributor is frequently at a very serious disadvantage in selling in competition with the local merchant. Consumer education is the hope of the ethical merchant. The latter awaits the day when the customer will know how to buy intelligently and to differentiate between the true and the false in advertising.

Advertising

It would be financial suicide for a house doing business with millions of customers to describe its wares in other than an honest manner. Immediate profit performance would be injured due to a return of the misdescribed merchandise, and future profits made impossible through loss of good will. Accuracy is assured in a number of way: First, the copywriter has the item before him as he prepares his advertising. This is supplemented by technical and merchandise comparisons reported by us. The Editorial Department which works very close to the laboratory, checks the proof for inaccuracies. Finally, feature pages are scrutinized again by the laboratory, in most cases the basis of the feature presentation being a laboratory report.

Sales Promotion

Important as checking and controlling the work of others is, there is a creative side to science as applied to merchandising that is just being realized. Laboratory work has too long remained a policeman with the promotive side badly neglected. It must be obvious that nowhere else can such fine sales promotional stories be developed as in a laboratory whose only passion is for accuracy and fact finding and, at the same time, being imbued with a sufficient sense of showmanship to present these facts in an interesting manner.

Development

To receive part of Ward's business means well managed companies with a national distribution, financial strength and capable of mass production. In no other way can adequate stocks be maintained at all points of distribution without excessive inventories. Such a plan gives the manufacturer an idea of what his production must be for six months, the life of a catalog, and at the same time gives him a flexible merchandising plan. If he sells under his own name and has a dealer organization, obviously

he cannot sell to Ward's for less without competing with himself. This works to the advantage of Ward's customers. Such items, identical in every respect except the name plate, are distributed to mail order customers at prices substantially lower than the advertised article.

There are many times when with our ear so close to consumer trend, we feel the need of something different, either in price, design or type. If nothing suitable is found available on the market, or the idea has unusual possibilities, that part of our staff of engineers and machinists whose job it is to work out the details, will develop the plans for manufacture. In such a manner has been born an unusually good low priced washer, vacuum cleaner, a new all-steel farm wagon, an absorption type refrigerator of conventional design to retail about $100, a low priced electric churn, etc. And for one entirely new development there are hundreds of constructive suggestions going back to manufacturers for modifications and improvements in design, material and workmanship.

With such a variety of work to be done that is unique, methods of analysis must be originated. A machine shop is maintained as an integral part of the department for building instruments, making modifications of apparatus and to maintain the standard testing devices at a high state of efficiency. Experience teaches us it is one of the most helpful units in the department.

Some pure research in chemistry and physics is also necessary. Gaps in the literature are rapidly filling up so that the standard handbooks can be turned to with increasing confidence, but much data remains to be collected on natural products.

...At the conclusion of Dr. Harvey's paper he exhibited a motion picture further elaborating upon the operation of his organization.

Following this there was musical entertainment and at the conclusion of this the meeting adjourned at eleven-fifteen o'clock.

SATURDAY MORNING SESSION
September 9, 1933

THE meeting convened in the Florentine Room, Congress Hotel, Chicago, Illinois, at eleven o'clock, William H. Cady presiding.

Chairman Cady: The meeting will please come to order.

We are late in starting this morning because the lantern is not yet ready. In order to avoid further delay, Professor Olney has consented to make the report of the Research Committee at this time instead of at twelve, as originally planned.

Before calling on him I have one or two announcements to make. In the first place, we are very sorry that we could not have Mr. Johnson's paper last evening. Owing to the lateness of the hour it was necessary to cancel that particular paper, but Mr. Johnson has very kindly consented to give it today if there is time. Perhaps we can put it in this afternoon.

Also, I have a telegram from Dr. Rose. As you probably know, Dr. Rose has hay fever, and is unable to be present at this Convention. He is extremely sorry to be away and has sent me the following telegram:

"Chemists and Colorists, Hotel Congress, Chicago:

"From the great canyons of the Southwest, whose colors have not faded in a million years, I send all of you my very best wishes for a great meeting. I regret exceedingly that I am not with you to greet each one of you at a convention meaning so much to the Association.

ROBERT E. ROSE, *President.*"

Is Mr. Gordon Pratt here? Mr. Pratt, will you stand, please? I have a letter for you.

I would like to introduce to you Mr. Gordon Pratt, member of the Society of Dyers and Colourists of England who is representing that Society at this meeting. (Applause.)

Now, gentlemen, we will hear the report of the Research Committee by Professor Louis A. Olney.

Report of the Research Committee

Professor Olney: It has been customary at the time of the annual meeting to have each sub-committee of the Research Committee report as to the work it has done during the previous year. For the reason that this is not the regular annual meeting and also because any such sub-committee reports would obviously be incomplete it was decided to omit them at this time. I will, however, speak very briefly in regard to the work of some of these committees.

There are, at the present time, twenty-two sub-committees and of this number six have practically completed their work. Nine more are well along with their projects, although by no means completed. Seven of them are of a continuous nature. Such for instance, as the Committee on Bibliography, Rayon, and Standard Methods of Chemical Analysis in Testing as Applied to the Textile Industry.

An additional sub-committee was authorized yesterday, at the Research Committee meeting, namely, one on the general subject of Mercerization. It is the intention to have the majority of the membership of this Committee from the South.

The Sub-committees on Fastness of Dyed Silk, Wool, and Cotton, have in general completed their work, but they are at all times open to suggestions as to how the methods already adopted may be improved.

The Committee on Light Fastness is at the present time occupied in checking the new German standards which consist of eight blue dyes, also the English stand-

ards, which have recently been approved by the British Society. And also a comparative study of the latest types of Fade-Ometer and Fugitometer.

The Sub-committee on Shrinkage of Textiles is continuing its work on the shrinkage of cotton material, and at the present time is planning to extend its work to materials made of fibers other than cotton.

The Sub-committee on Rayon is continuing its excellent work which it has been doing for several years, and is now engaged in a study of shortcut tests for determining certain properties and causes of faults observed in rayon yarns and fabrics, with the object in mind of compiling a group of the most satisfactory methods.

The Sub-committee on Analysis and Standardization of Sulfonated Oils is making an exhaustive and painstaking study of this subject, and expects to complete the main portion of its work some time during the coming year.

The Sub-committee on Standard Methods of Chemical Analysis and Testing as applied in the textile industry is now well organized and the coming year should see its work well advanced. It is the objective of this Committee to bring together for publication later in book form the most approved methods for the use of the textile chemist and colorist.

We have four full-time research associates working under the direction of the Research Committee at the present time. Two at the Bureau of Standards in Washington, one at the Lowell Textile Institute, and one at Brown University. Their work is being supported largely through allocations from the Textile Foundation. At the present time, four distinct research products are being supported by the Textile Foundation. They are as follows: First, a study of the carbonization process; second, wool scouring, including combinations of soap and alkali with wool, and their removal from wool; third, textile dyeing, the relationship between the quantity of dye absorbed, and the charges on the fiber and on the dye; fourth, a fundamental study of printing pastes, more especially vat dye printing pastes.

The Research Committee meets upon an average of eight times a year, and the meeting held yesterday was the eighty-third meeting.

In closing this report, I should like to say just a word about the new type of membership, namely, sustaining membership, which has been voted by the Association. The object of this membership is to secure additional funds for the promotion of the work of the Research Committee, and beginning this Fall, every member will have an opportunity to become a sustaining member by paying $10 additional dues. In this connection, I might add that in the early days of the Research Committee, we solicited funds and one year got as high as $2,000 through subscription from the members. We have not done that for several years, and we thought sustaining membership would be a more satisfactory and uniform method of accomplishing a similar result. Thank you! (Applause.)

Chairman Cady: Is Dr. Harris here?

Gentlemen, we will now have the first paper on the program for today. Dr. Milton Harris, Research Associate of this Association, will speak on "The Carbonization Process: A Study of the Effect of Sulfuric Acid on Wool." Dr. Harris!

. . . Dr. Harris presented his paper which will be submitted later to THE DYESTUFF REPORTER . . .

Chairman Cady: Is there any discussion of Dr. Harris' paper?

Is Mr. Clayton here? I am afraid that Mr. Howard D. Clayton, who was to give the next paper has not yet arrived. We will pass on to the next paper.

Is Mr. Smolens here? Mr. H. G. Smolens, of the Buffalo Electro-Chemical Company will talk on "Peroxide Bleaching, a Report of Progress." Mr. Smolens: (Applause.)

(See next page)

Proceedings of the American Association of Textile Chemists and Colorists

Peroxide Bleaching

A Report of Progress*

By **H. G. SMOLLENS**
Consulting Engineer, Buffalo Electrochemical Co., Inc.

THE past six years have seen greater advances in peroxide bleaching in this country, than did the preceding thirty years. This is due, partly, to the extra efforts made by the manufacturers of the 100 volume peroxide to help bleachers to apply them properly; partly, to the arousing of a real interest in peroxide bleaching among finishers of woolens, silks, cottons; partly, to the unprecedented circumstances under which the entire textile industry has been operating.

These advances have been accompanied by considerable improvements in the whiteness and general quality of the bleached textile and by marked reductions in the unit costs of these superior bleaches.

Now, there is a possibility that we are fairly close, in many lines, to the limits of both the quality and cost of peroxide bleaching, with the equipment that we are using. Further advances may depend on the introduction and use of new equipment, specifically designed for peroxide bleaching. This equipment may be the outcome of the many improvements being made in the construction of the present type of machinery. Or, it may be the development of entirely new types of apparatus depending for its design and operation, largely on the successful conclusions drawn from new ideas of peroxide bleaching that are constantly undergoing investigation.

There are already available sufficient experimental and practical data and experiences in peroxide bleaching of all kinds, to work out a satisfactory bleaching process for any textile, under almost any combination of local conditions. Or, it is readily determinable that definite changes made in these conditions, will allow satisfactory bleaching. This means, that in 1933, no one can push aside peroxide bleaching with the 1926 remark "It will not work here."

To give an actual listing of the accomplishments of peroxide bleaching during the past six years, would be an injustice to the manufacturers and finishers of textiles, whose whole hearted cooperation was so essential to the success of our efforts and who have, we hope, profited accordingly. However, it may not be an abuse of confidence on our part, to bring out the following contracts between peroxide bleaching as done in 1933 and peroxide bleaching as done in 1926.

Wool Raw Stock

At least twenty million pounds of wool raw stock are being bleached annually in the continuous scouring machines, at an average cost of less than ½c. per lb. over the usual cost of scouring. This process, in spite of its little advertised success, is not as perfect as it can and will be. There are still difficulties in obtaining continuously uniform shades of the bleached wools and in the maintenance of the quality of the bleached stocks from the standpoint of the spinner, the dyer and the finisher.

In 1926, wool raw stock that was to be bleached, was first scoured, washed and usually dried. It was then bleached by an overnight submersion in a peroxide bleaching solution, after which it was washed and dried. All of this processing cost, in chemicals alone, about 3c or 4c per lb. plus the cost of losses in carding and spinning due to brittleness of the bleached fibers, necessary reduction in running speeds, etc.

Worsted Top

A fairly large poundage of worsted top is being very well bleached in the newer top dyeing machines, at a chemical cost of about 2c per lb. The entire process of bleaching, washing and drying takes less than six hours. Further improvements in top bleaching, which will still better the quality and reduce the cost of top bleaching, will soon be ready for introduction.

In 1926, worsted top was bleached in a manner similar to that outlined for wool raw stock, at a chemical cost of 5c or 6c per lb. to which was added the cost of a recombing of the bleached top which was expensive as well as troublesome.

Knitting Yarns

The bulk of wool and worsted outerwear knitting yarn is being so well bleached today, from the standpoints of color, strength, loftiness, winding and knitting qualities, that there is nothing in common, chemically, between a 1933 bleach and a 1926 bleach. However, only a few of the wool and worsted yarn bleachers, are making any profit on their work at the present prices for bleaching. These do so only because they use their peroxide bleaching liquors continuously and control them very accurately

* Presented at Saturday morning session, Annual Convention, Chicago, September 9.

both in alkalinity and peroxide concentration. This not only makes possible low chemical costs of about 1½c per lb. of yarn, but also allows close matching of whites, which is quite important to knitters. Aside from this, there has been no real improvement in the general processing required for the bleaching of wool and worsted yarns. The handling costs, especially, are too high. We are working on this problem and hope that it will be solved soon.

Wool and Worsted Piece Goods

Several years ago, such a marked improvement was made in the bleaching of wool piece goods, that it was no longer necessary to sulphur them, after the peroxide bleach, for good marketable whites. Now, a two or three hour run in the proper peroxide bleaching solution, followed by a souring or a bluing, depending on the shades desired, gives a result at least the equivalent to that obtained in the following process used in 1926. Then, the cloth was run for several hours in a peroxide solution, opened up and submerged overnight in the same or a fresh solution, sewed together again in the morning and run for several hours more. After this, the cloth was washed, squeezed, hung up in the sulphur house for a good smoking, and finally aired to remove the sulphur dioxide fumes.

Wool Felt Hats

Until 1930, practically all of the white wool felts sold in this country, came from either France or Italy. Today, our American white felt hat body is so superior in every respect to the former standards, that hat manufacturers in Europe are sending men over to try to find out what we are doing.

Silk Bleaching

In the silk finishing field, probably the largest problem that has been solved since 1926 is that of bleaching tussah silks without, at the same time, tendering them. This was accomplished by first finding out how to prepare tussahs for the peroxide bleach; then by determining the limits of total safe alkalinity that could be used at the usual bleaching temperatures. Now we are bleaching tussahs for colors in two hours and for whites in three hours. Recently a new type of process for bleaching tussahs has been tried out, with very promising results.

There have also been worked out excellent new bleaching processes for silk rayon mixtures, and especially for silk celanese mixtures.

Cotton Bleaching

Now we come to cotton bleaching with peroxide, in which there are still many differences of opinion. To even outline the advances in the peroxide bleaching of cottons during the past six years, would require at least a whole day, aside from the discussions that might arise.

It was acknowledged, even in 1926, that colored yarn soft cotton fabrics, such as terry towels, could be bleached fairly well with peroxide, if one were lucky, but who, at that time, ever expected to see sheetings, both fine and coarse, print cloths and sheer fabrics, peroxide bleached, in volume. A few bleachers agreed that some types of knit cotton tubing, for underwear, could be peroxide bleached, if no account were taken of costs, but when drills, napped goods, sateens and broadcloths were mentioned, there was unanimous laughter. Now, 45,000 yard lots of colored shirtings are run into a kier for peroxide bleaching and no one considers it a matter of undue interest. Or 35,000 yard lots of cloth that is to be made into washable window shades, are run in and out of peroxide bleaching kiers without a second thought, except when there may be trouble in packing the bleached cloth in the former standard cases, because of its extra weight.

Cotton yarn bleaching is another good example of peroxide bleaching progress. A majority of the most particular knitters are now demanding peroxide bleached yarns for their higher speed machines, either in white or in light colors. Most of these yarns are bleached in the package dyeing machines. The elastic trades and lately the lastex makers have standardized almost entirely on peroxide bleached yarns. Even in thread yarns, including saks and egyptians, it has been found that a peroxide bleach is quite desirable, especially for flat lock sewing machine work.

About four years ago one very prominent lace curtain fabric manufacturer discovered that the peroxide bleach would and did loosen the oily graphite that is on all woven laces, and that a simple washing, after the bleach boil, cleaned up such goods better and quicker than had ever been thought possible.

All of this would indicate that the peroxide bleaching of cottons is already a cut and dried process, but such is far from the case. There are still too many problems that not only have not been solved, but are encountered too frequently for comfort. The following examples are illustrative.

We believe that some treatment of cotton piece goods prior to piling them in the kier, is not only desirable but also economical. However, we do not know yet, just what kind of treatment is the best for the final peroxide bleaching. Advice has been obtained from experienced bleachers, dyers and finishers, but their opinions are so contradictory that we get nowhere. Furthermore, the same preparation of cloth for apparently the same dyeing and finishing processes, produces different results in different plants.

Water plays an important part in the success of peroxide bleaching, possibly more important than we realize. However, we do not yet know just what to do about water, as we have troubles with spots, resists, etc. in plants that have filtered and softened water, while plants with apparently undesirable water are doing very successful peroxide bleaching, on similar fabrics.

It is not fully known or understood that a preboil is necessary or desirable for many cotton materials, such as knit goods, for example. It has not yet been determined that a long caustic boil with high concentrations of chemicals is better for an after peroxide bleach than a short boil with lower chemical concentrations. Even the value of pressure in the preboil is still in the argumentative stage.

Coming to the chemicals, aside from the peroxide itself, that are ordinarily used in making up the bleaching solutions, we must also confess a few uncertainties. Of course, we take it for granted that the hydrogen peroxide itself, is quite essential. We also think that silicate of soda, especially the one with the ratio of 1.0 Na_2O to 2.5 SiO_2, is almost as essential. Besides these we have a free choice of caustic soda, soda ash, borax, the phosphates, other silicates of soda and innumerable oils, wetting out materials, general assistants, special assistants, etc., any or all of which may have their places.

On the basis of the unknowns, only a few of which have been mentioned above, one can easily wonder how it has been possible to work up to the quality and quantity of peroxide bleached cottons that are produced daily. The following is one explanation.

The first necessity was a peroxide that we could have implicit confidence in, under any conditions. This was met by the introduction of the 100 volume peroxides, in 1927. With them as a foundation, we knew that any process which did not work as expected, could not be blamed on the instability, the non-uniformity or the general uncertainty of our peroxide. So, we examined the other chemicals, the equipment, the materials being bleached, their preliminary treatments, etc. This was done in a roughly scientific manner and on a practical enough scale to arouse the curiosity and interest of the bleacher. He, in turn, applied his practical experience to our chemical shortcomings, with the most gratifying results. Then came the battle with the dyer and the finisher, but we already had on our side the bleacher, who is quite expert, as a result of much experience, in getting around dyers and finishers, so the conflict was not very long or hard. Finally, the market likes peroxide bleached cottons more and more every season.

If, with the handicaps that we had six years ago, we have been able to bring peroxide bleaching of cottons to its present status, is it not logical to expect even greater things, as we gradually reduce or do away with these handicaps?

It is not so long ago, that we came to the conclusion that hydrogen peroxide bleaching is particularly adaptable to the ideal which all of us interested in textile finishing have, viz. the continuous process. Surprising as it may seem, on account of the lowness of the costs involved, more thought and action along this line has been applied to cotton finishing than to either wool or silk. Several years ago, a continuous cotton goods boiling machine, which was intended also to do peroxide bleaching, was tried out on a fairly large scale, but we did not know enough, at the time, to make it successful. However, there were obtained a few ideas of continuous bleaching which we think have been worked out. But, we are not yet able to get around the kier boil, either for quality of work or for costs of operation. As soon as we do, there will probably be many discoveries, that, although incidental to the discarding of the kier and its present usage, may be of tremendous importance to the entire cotton finishing industry.

There will soon be available, we hope, newly developed peroxide bleaching processes for rayon yarns or fabrics and for linen yarns and fabrics. These have always been more or less of a blank for peroxide bleaching.

A great deal of work has recently been done in the peroxide bleaching of such fibers as leathers, horsehairs, bristles, etc., which, although not textiles, strictly speaking, are closely related to them.

If peroxide bleaching makes the same relative progress during the next six years that it has made during the past six years, it will be an accomplishment of the textile finishing industry that we, as members of an association of textile chemists, may all be proud of.

Discussion

Chairman Cady: Is there any discussion of this paper?

Mr. Schroeder: In the bleaching of thread yarn, is that done in skeins or in warps?

Mr. Smolens: In skein or packages.

Mr. Schroeder: Not in warps?

Mr. Smolens: Not as far as I know.

Mr. Schroeder: Are you a believer in boiling it out in caustic soda?

Mr. Smolens: What type of yarn? It depends on the type of work that is going to be done with the thread, on the bleaching equipment, and on the local conditions.

Mr. Schroeder: When you have found tendering of yarn, is that liable to appear in the boil-out or in the bleaching?

Mr. Smolens: I would say in the bleaching. Just offhand I would say that tendering of yarn occurs in the bleaching, unless you are pressure boiling it.

Mr. Schroeder: No, we do not pressure boil it.

Mr. Smolens: Then I would say that tendering of the yarn occurs mainly in the bleaching.

Mr. Schroeder: Is it due to too small an amount of alkali or too large an amount?

Mr. Smolens: That is a difficult question to answer.

Chairman Cady: Is there any further discussion of this

paper? If not, we will interrupt the technical program for a moment and listen to Captain John W. Gorby, of the Department of Promotion of the "Century of Progress" who has a message for us.

. . . Captain Gorby addressed the meeting, extending a welcome to the city of Chicago, and explaining some of the features at the Fair in which the members of the Association might be particularly interested. . . . (Applause.)

Chairman Cady: We shall have to make a slight change in the program because the lantern is out of commission, and so we will call next upon J. G. Stott, Jr., of the Ciba Company who will talk on, "A Century of Progress in the Wallpaper Industry."

. . . Mr. Stott presented his prepared paper. . . .

A Century of Progress in

The Wallpaper Industry*

By **J. G. STOTT, Jr.**

Ciba Company, Inc.

MY INTENTION is to give you a short resume of one of Chicagoland's prominent industries. In doing so, I am going to stray from the textile field—but not too far.

Gorgeous and elaborately woven silks, embroidered draperies and tapestries covered the walls of the early Greeks and Romans. Later on, they became a real necessity in the homes of the Franks and Burgandians. Their walls were cold, unattractive and crude, and where the climate was most severe, these various devices were successful in creating an atmosphere of comfort and warmth. As the materials were very expensive, due to the long labor of weaving by hand, only the wealthy could possess them.

It was found that paper, properly colored and designed, fulfilled all requirements, so the vogue spread very rapidly. Hand-painted and block-printed papers were the first developed, then flock papers were introduced, and finally the roller-printing—which closely resembles the textile printing.

France, England and Holland all claim the honor of originating the first European wallpaper. The earliest wallpaper of which we have any record was hand-painted. There is a record of Louis XI, in 1481, paying twenty-four Francs for fifty rolls of paper in blue. The oldest wallpaper that has been preserved to us was found in England, being a copy of an early damask.

Chinese paper had its influence in the 17th century, having introduced wallpaper with gay designs. Their patterns were of three kinds—landscape, bird and flower, and scenes of domestic life. In all these patterns they were following a long established tradition in Chinese painting.

Not until the second quarter of the 18th century was wallpaper manufactured in this country. Philadelphia claims the honor of having produced the first—the maker being Plunket Flaeson, an importer. In 1765, John Rugar began the manufacture of wallpaper in New York, and in the eighties another factory was started in Boston. The early American wallpapers were highly colored, elaborate scenes, and the quality was far from equal to the finest papers being imported at that time. The cost of furnishing one room during this early period was about one hundred and forty dollars.

A typical example of the first American wallpaper was found in a house in Hampton Falls, New Hampshire. The pattern is a complete hunting scene, showing the deer, dog and hunter with his horn. Scenes of foreign cities and other places of interest were popular. One Oriental pattern depicts the entire process of making tea.

*Presented at Saturday morning session, Annual Convention, Chicago, September 9.

Wallpaper is primarily printed on a white or ecru stock, consisting chiefly of wood pulp and bleached sulfite. The finest grades are printed on rag stock.

The colors used today consist of pigments, metals, oil colors occasionally, and laked fast to light aniline dyes. These colors are made into a paste and, with the addition of glue, are made to adhere to the paper. The wallpaper mills of today have recently added another attractive feature—namely, making papers fast to a sponge cleaning with soap and water.

The artist first draws the actual design and paints in the necessary colors. It is then sent to the block cutting department where a sketch is made from this design and transferred to a wooden roller. The outline of this design is built up by means of brass strips about one-quarter of an inch in height. Where large surfaces have to be printed, it is necessary to insert piano felt. For this certain design, there may be one roller or twelve, depending upon the number of colors used in the design.

The actual printing is a very simple mechanical process. It is done on a cylinder press. The paper, in a large roll about 1,500 feet long, passes over several small wooden rollers that act to keep the tension even under the drum. As the paper passes around the drum, a series of cylinders roll over the surface of it and print the various designs. No two prints ever over-lap—they fall side by side. The modern machine will have one to twelve color boxes. In the troughs containing the colored pulp is placed an agitator, which keeps the color mixed, and from this trough the color is fed to the cylinders by means of a sieve cloth. This porous cloth is stretched over two or three rollers and forms the contact with the cylinder. There are times when one color is printed over another; then the paper has first to be dried between operations. The drying is done by allowing the paper to droop in long loops over horizontal bars on an endless chain-carrier in a drying room. In this room are steam pipes and a fan which blows the warm air on the slowly moving paper. As the paper comes off the drying racks, it is reeled into a large roll. It is then rolled into the regular market lengths and labeled ready for shipment.

Flock paper was first introduced by France in the early 17th century. Le Francois, of Rouen, was among the first to imitate silk weaves for wall hangings. Rouen was at that time a very great center of the silk weaving industry, so it was an easy matter to obtain all the silk waste necessary for his work. This paper became very popular; its manufacture was greatly improved in England and, for a century, that country reigned supreme in its production. Of especial interest is the fact that it was made to resemble damask, velvet and silk weaves of that period. This style of paper is now manufactured on the same type machine as previously described. The design is first printed with glue, and, while still wet, powdered wool or silk is sifted upon it.

There are a few kinds of paper that are dyed in the pulp—oatmeal and crepe being the most common.

The wallpapers of today run from the modernistic to the dainty ones of my lady's boudoir. They are selected to blend with the furniture, paneling or color scheme of a room. There are papers printed which are suitable for only the kitchen, the nursery or the halls. Each of these fills a particular need in beautifying and making the home more attractive.

The classes of wallpapers which are termed varnished tiles and sanitary engraved papers are printed by the rotogravure process, which is similar to the calico printing method. In these cases, pigments are used in combination with varnish for binding the color to the paper.

In closing, I wish to thank Arthur Bowden, of the Henry Bosch Company, for his kind cooperation in helping me collect the necessary data. Mr. Bowden has offered to show anyone through their plant who has the time and opportunity to make the visit, while in Chicago. He will also be glad to answer any question that you may care to ask.

I thank you.

Discussion

Mr. Stott: I am very sorry to say Mr. Bowden is not here or he would be delighted to answer any questions you might have to ask. (Applause.)

Chairman Cady: Is there any discussion of this paper?

We will pass on, then, to the next paper. Dr. Walter M. Scott will give a paper on, "A Comparison of Methods for the Estimation of Small Amounts of Copper in Textiles." Dr. Scott!

. . . Dr. Walter M. Scott then presented his prepared paper as follows:

(See next page)

Proceedings of the American Association of Textile Chemists and Colorists

A Comparison of Methods for the Determination of Small Amounts of Copper in Textiles*

By WALTER M. SCOTT, Ph.D.
Gustavus J. Esselen, Inc., Boston, Mass.

THE determination of small amounts of copper in textile materials is a subject which has become increasingly important in the last few years, because of the ever expanding use of textiles in connection with rubber or other plastic coatings. It is well known that the presence of any appreciable quantity of copper will tend to accelerate the decomposition of a rubberized fabric, although there has been considerable disagreement as to the permissible allowance for copper.

In order to shed some light upon the order of magnitude which these particular copper analyses have to deal with, it will be necessary to give a brief resume of the controversy between the rubberizers on the one hand and the textile manufacturers on the other. Because of their desire to be entirely on the safe side, the rubberizers attempted to set a limiting specification of 0.001% as the maximum quantity of copper to be allowed in fabrics which were to be rubberized. This specification was actually adopted as tentative by the A. S. T. M. in 1931 and will be found among their list of tentative standards under the designation D334-31T, "Tentative Specifications and Test Methods for Cotton Goods for Rubber and Pyroxylin Coating." The same specification was incorporated by the U. S. Bureau of Standards as a part of their commercial standard, C. S. 32-31, "Cotton Cloth for Rubber and Pyroxylin Coating."

The textile manufacturers, on the other hand, maintained that it was impossible to keep the percentage of copper in cotton textiles as low as 0.001%, even though special precautions were taken all through the dyeing and finishing processes. One manufacturer of tapes, bindings and narrow fabrics went to the trouble and expense of making a large number of copper analyses both on the raw cotton and on the goods in the various stages of manufacture in his plant. The analyses were checked by two different methods in order to insure the accuracy of the results. It was found that raw cotton itself contained over 0.001% of Cu. In fact it varied from .0013% to .0016% in copper content. Because of the strong affinity of cotton for copper, it was not commercially feasible to lower this percentage during the process of manufacture, although with due precautions it was possible to spin, weave and dye the cotton without any appreciable increase in the copper content.

The first desire of the mill men was to have the maximum copper standard set at 0.005%. As a matter of fact many thousands of yards of cotton piece goods have been processed under this higher specification and have been used in rubberized raincoats without any serious complaint or rejection due to the deterioration of these raincoats in actual usage.

The writer has been informed by the chemist of a large rubber company that the amount of copper which can be safely used in a rubberized fabric depends largely upon the method of vulcanizing, as well as the kind of rubber mixture and accelerators incorporated in it. It is the opinion of this chemist that no buyer has need to worry about sources of supply that will meet a specification for copper of 0.003% maximum. And it is this latter figure that has finally been pretty generally accepted both by the textile manufacturers and the rubberizers as a compromise between their original demands. In the 1932 Year Book of our Association it is stated on Page 155 that the limit of 0.003% for copper has been definitely adopted by the Rubberizers' Standards Committee.

In the analysis of fabrics for copper, if we start with a five gram sample which contains 0.003% of copper, the total amount of copper to be determined will be 0.00015 gm. or 0.15 mgm. When the quantities are as small as this it is somewhat more convenient to use the unit of micrograms (expressed by the symbol λ), one microgram being equipment to one-thousandth of a milligram or one-millionth of a gram. Thus it will be seen that we are dealing with amounts of copper in the neigh-

*Presented at Saturday morning session, Annual Convention, Chicago, September 9.

borhood of 150 λ if our fabric sample is about 5 grams, or 30 λ of copper in a one gram sample. We shall refer to these amounts of copper from time to time in the following brief history of the methods which have been proposed for the determination of small amounts of copper.

When dealing with normal amounts of copper it is usually determined quantitatively either electrolytically by deposition on a platinum electrode or volumetrically by the liberation of an equivalent amount of iodine and the titration of this iodine with a N/10 standard solution of sodium thiosulfate. The conditions under which these two methods of analysis could be most satisfactorily run were discussed very thoroughly by Peters (1) in 1912 under the title, "Sources of Error and the Electrolytic Standardization of the Conditions of the Iodide Method of Copper Analysis."

The electrolytic method has never been considered suitable for the determination of very small quantities of copper, although Okac (32) in 1932 described the conditions suitable for the deposition of 200 to 5,000 λ of copper in a total volume of 6 to 7 c. c. If a 10 gram sample of fabric was used there would be about 300 λ of copper and this would fall within the range of this method. However, the writer is not aware that any serious consideration has been given to the determination of copper in textiles by the electrolytic method.

The volumetric method, on the other hand is capable of being adapted to the determination of relatively minute amounts of copper, by using a N/100 or N/1,000 standard solution of sodium thiosulfate, and has been recommended for this purpose by a number of investigators. It has been tentatively adopted as the official method for the analysis of copper in foods by the Association of Official Agricultural Chemists, and a detailed description of the procedure is given in the Third Edition (1931) of their "Official and Tentative Methods of Analysis" (36). Walker (25) in 1930 published a detailed discussion of the application of this method to the analysis of food products and outlined the most satisfactory procedure in order to minimize errors. Kirchof (30) applied this method to the determination of copper in fabrics and rubberized materials, and several months ago it was adopted as a tentative standard method by Committee D-13, the Textile Division of the American Society for Testing Materials (38). A more detailed discussion of the advantages and disadvantages of this method will be given later in this paper.

Recently an even more sensitive titration method has been developed for the determination of copper in organic materials. The titration agent in this case is a standard solution of nitrosochromotropic acid. The application of this method to the determination of copper in the organs of the body was described by Cherbuliez and Ansbacher (18, 19) in 1930. Additional researches along this line were published by Andrianoff and Ansbacher (16) in 1930, and a detailed description of the best method of procedure for the analysis of foods was given by Ansbacher, Remington and Culp (26) in 1931.

In brief, this method is based on the fact that the nitrosochromotropic acid "forms with copper in a weakly ammoniacal solution a compound which has a very intense permanganate color, resulting from the union of one cupric ion with two molecules of nitrosochromotropic salt. As soon as an excess of the yellowish brown nitroso dye is present, the permanganate color changes to brown."

This color change serves as indicator and it is possible (according to the authors) to estimate quantities of copper as small as 2 to 3 λ with an accuracy of 0.5 λ. In so far as the writer knows, there is no published record of the application of this titration method to the determination of copper in textiles but it certainly seems worthy of careful investigation.

By far the largest number of investigators have turned their attention to colorimetric methods for the analysis of copper when quantities of less than 200 λ were involved.

Our attention is first called to the potassium ferrocyanide method, which depends upon the development of the red copper ferrocyanide color, because this has been in use for a longer period of time than any of the other colorimetric methods. A very good discussion of this method was given by Pritz, Gillaudeau and Withrow (2) in 1913. They record that Carnelly in 1875 claimed that ferrocyanide will detect one part of copper in 1,000,000 (i.e. 5 λ in a 5 gm. sample.) Bradley, in 1906, found that potassium ferrocyanide could detect one part of copper in 100,000 (i.e. 50 λ in a 5 gm. sample) but not much less than this. Ammonium nitrate increases the delicacy of the test. Iron interferes and must first be removed.

A quantitative determination of copper by this method is made by comparing the depth of color developed by the unknown, with the color of standards containing known amounts of copper, in combination with a definite amount of potassinum ferrocyanide. The application of this method to the determination of copper in fabrics was discussed by Rupp (22) in 1930, and by Trotman (34) and Holst (29)) in 1932. This was the preferred method of the manufacturers of rubber and pyroxylin coated automotive fabrics in 1931 and is the present tentative method of our Association. A detailed description of the procedure is given in the 1932 Year Book of the A. A. T. C. C. (37) on Page 155. The advantages and disadvantages of this method will be discussed later in this paper.

Another well known reaction which is applicable to the colorimetric determination of small amounts of copper is the yellow color which it develops with potassium ethyl xanthate. This method was used by Supplee and Bellis

(6) in 1932 for the determination of the copper content of cow milk, and they claimed that it was reliable to 5 λ. Smith (24) in 1930 applied this method to textiles and apparently obtained quite satisfactory results. However, at the last annual meeting of our Association, Chase (35) showed that the intensity of the yellow copper ethyl xanthate decreased rather rapidly if the concentration of copper was less than 60 λ, and increased if the concentration was more than 60 λ. At about 60 λ the color was quite permanent.

Drabkin and Waggoner (20) claim that the potassium ethyl xanthate method is quite reliable if the pH of the unknown and the standard solutions is adjusted to 3.8 by the use of a phthalate buffer. Ansbacher, Remington and Culp (26) made a very careful study of this method in comparison with several others and they state that it tends to give high results, the error being least if from 100 to 200 λ of copper are present. Iron interferes to the extent that about the same color is obtained with the xanthate from 50 λ of iron as from 12.3 λ of copper. A further discussion of this method will be given later in this paper.

A rather novel color reaction of copper was described by Spacu (3) in 1922. He discovered that a compound insoluble in water is formed by adding a neutral solution of a thiocyanate and a few drops of pyridine to a neutral solution of a copper salt. This compound has the formula $Cu\ Py_2\ (C\ N\ S)_2$. He claimed that the reaction is sensitive to one part of copper in 300,000 and that its sensitivity can be increased to one part in 800,000 by shaking the mixed solutions with several drops of chloroform which assume an emerald-green coloration. Sapcu (4, 5) adapted this reaction to the gravimetric determination of copper but Biazzo (10) in 1926 used it as a colorimetric method for the determination of copper in preserved vegetable products and it has since been known as the Biazzo method. Elvehjem and Lindow (13) and Schönheimer and Oshima (14) in 1929 applied this method to the determination of copper in biological materials, while Gebhardt and Sommer (27) applied it to dairy products with some slight modifications. Chalk (17) adapted it to the determination of small amounts of copper in the presence of iron, and Drabkin and Waggoner (20) showed that the interference of iron can be overcome by adding to the copper solution 3 to 10 drops of a 4% solution of sodium pyrophosphate before the addition of the potassium thiocyanate and pyridine.

Ansbacher, Remington and Culp (26) in 1931 found that the Biazzo method is most reliable for 50 to 150 λ of copper, but that it tends to give results which are slightly low. Earlier investigators of this method claimed that 10 λ of copper could be determined with a maximum error of 5%. There is no record of the application of the Biazzo method to the determination of copper in textiles.

An extremely sensitive color reaction for copper was described by Callan and Henderson (11) in 1929. They found that sodium diethyldithicarbamate,

$$(C_2H_5)_2\ NCS_2\ Na$$

gave a brown precipitate with copper. They claimed that this reagent would detect one part of copper in 100 million parts of water (i.e., 0.05 λ in a 5 gm. sample). Haddock and Evers (28) applied this method to the colorimetric determination of minute amounts of copper in the presence of iron by extracting the color with carbon tetrachloride, while McFarlane (31) used amyl alcohol to extract the color. Chase (35) made some preliminary experiments in the analysis of textiles and he states that for quantities of copper over 10 λ it is not necessary to extract the color with amyl alcohol, but that for lesser quantities of copper the extraction tends to increase the intensity of the color and thus make easier the comparison with standards. Ansbacher, Remington and Culp (26) found that the carbamate reagent is particularly applicable to solutions containing about 50 λ of copper with a maximum error of not over 1½%. This is a method which seems worthy of further investigation by the textile chemists.

A number of other sensitive color reactions for copper have been described in the literature and will be briefly mentioned at this time because of the possibility that they might at some time be adapted to the determination of small amounts of copper in textiles.

1. Copper-Benzoin Oxime (cupron) gives a green precipitate with cupric ions. It is sensitive to 100 λ according to Strebinger (7).

2. Benzidine-Thiocyanate—a deep blue precipitate results when to a dilute copper solution are added 3 to 4 drops of a dilute solution of potassium thiocyanate and a few drops of a 1% solution of benzidine in alcohol. Sensitive to 1 part of copper in a million according to Fleming (8).

3. Toluidine-Thiocyanate—Spacu (9) in 1925 stated that if a very dilute copper solution was treated with a few drops of potassium thiocyanate and not more than 2 drops of a freshly prepared solution of toluidine in alcohol, a characteristic blue precipitate results.

4. Benzidine-Iodide—Spacu (9) also discovered that if a dilute copper solution is treated with 2 c. c. of potassium iodide solution and then with 3 drops of a freshly prepared 1% solution of benzidine in alcohol, a flocculent dark blue precipitate results.

5. Dimethylglyoxime—In 1929, Clarke and Jones (12) added to the faintly acid copper solution, 1 gm. of ammonium persulfate, 1 c. c. of a saturated solution of dimethylglyoxime in alcohol, 0.5 c. c. of a 0.5% solution of silver nitrate and 2 c. c. of a 10% aqueous solution of pyridine. A reddish violet color results. One part of copper in 10 million of water can be detected by this

reaction. In 1930 Kolthoff (21) confirmed this observation and found that a trace of periodate could be substituted for the persulfate.

6. Ethylene Diamine-Potassium Mercuric Iodide—Spacu and Suciu (15) obtained a characteristic precipitate when a neutral or slightly acid solution of copper was treated with ethylene diamine and potassium mercuric iodide in excess.

7. P-Dimethylaminobenzylidene-rhodanine was suggested as a reagent for testing for copper by Kolthoff. (21)

8. Direct Green B—According to Sisley and David (23), when copper salts are added at 90° C. to a solution of Direct Green B, the solution changes from blue-green to violet-pink. The solution should have a pH between 6 and 9 and the reaction is sensitive to 100 λ of copper per liter. No other metal gives a violet-pink color.

Having given a brief summary of the numerous methods which have been proposed for the detection and estimation of very small amounts of copper, this paper will conclude with a more detailed discussion of three of these methods: namely, (1) colorimetric with potassium ferrocyanide, (2) colorimetric with potassium ethyl xanthate and (3) volumetric with sodium thiosulfate. The writer is chairman of Sub-Committee B-4 on Bleaching, Dyeing and Finishing, of Committee D-13 of the A. S. T. M. This sub-committee has been investigating the three methods mentioned above and during the past year extensive series of experiments have been carried out by several interested laboratories. A description of these experiments and a summary of the results will be given in the following paragraphs.

In order to make all of the results strictly comparative, each laboratory used the same method of preparation for the standard samples used in checking the different analytical procedures. Pieces of cotton cloth weighing 5 grams each were impregnated with a standard copper solution containing 100 λ of copper, equivalent to 0.002% of copper on the weight of the goods.

In the first place a comparison was made of two methods for disintegrating the fabric and getting the copper into solution. In the so-called ashing method the sample was placed in a small evaporating dish and heated with a low flame from a Bunsen burner until only a carbonaceous skeleton remained. Then a few drops of nitric and sulfuric acids were added and the heating continued until all the carbon and tarry residue were burned off. The ash was then dissolved in conc. hydrochloric acid.

In the digestion method the sample was placed in a Kjeldahl flask, to which was added 25 c. c. of conc. nitric acid, 25 c. c. of conc. sulfuric acid and 5 grams of potassium sulfate. Heat was strongly applied to this mixture until the fabric was completely disintegrated. The clear melt was then taken up with distilled water.

The determination with ferrocyanide was carried out as follows:—the copper solution was made alkaline with ammonia, filtered to remove the precipitated ferric hydroxide and evaporated until neutral to litmus. Then in a 50 c. c. Nessler tube, ½ c. c. of 0.5% solution of potassium ferrocyanide was added and the total volume made to 50 c. c. with distilled water. The color was compared with the color of known amounts of copper treated with the same amount of potassium ferrocyanide.

For the potassium ethyl xanthate determination the copper solution was made alkaline with ammonia and then just acid to litmus with dilute sulfuric acid. Five c. c. of 10% phosphoric acid and 3 c. c. of 0.1% potassium ethyl xanthate were added and the color compared in 50 c. c. volume with the colors of known standards treated with equal proportions of the same reagents.

The volumetric method requires a preliminary removal of iron by adding ammonium hydroxide in slight excess and filtering. To the filtrate was added sufficient dilute acetic acid to make it faintly acid, then 5 grams of potassiumiodide and 1 c. c. of a 1% starch solution. The titration was carried out immediately, using freshly prepared N/1,000 sodium thiosulfate.

Each laboratory carried out the three analytical procedures on samples which were disintegrated by both the ashing and the digestion methods. At least four duplicate determinations were made in each case and the results averaged. In all cases the results were corrected by a blank run on the untreated cotton cloth with the same reagents and procedure. The results are summarized in Table I.

Table I.

Results of the Analyses of Standard Samples of Cotton Fabric. Each Sample Weighed 5 grams and Contained 100 λ of Copper

1. Colorimetric with Potassium Ferrocyanide

Lab. No.	Average Analysis Ashing λCu	Digesting λCu	Error Ashing λCu	Digesting λCu
1.	100	100	* 0	* 0
2.	100	87.5	* 0	— 12.5
3.	90	102.5	—10	+ 2.5
4.	90	88	—10	— 12

*These results are not significant because the calculations were not carried far enough to indicate the variations from 100λ of copper.

2. Colorimetric with Potassium Ethyl xanthate

Lab. No.				
1.	120	113.5	+20	+ 13.5
2.	87.5	87.5	—12.5	— 12.5
3.	....	....	.....	.....
4.	114	110	+14	+ 10

3. Volumetric with Sodium Thiosulfate

Lab. No.				
1.	101	101.5	+ 1	+ 1.5
2.	...	.....	....	.....
3.	80	100	—20	0
4.	104	111	+ 4	+ 11

It will be seen that as far as accuracy of results were concerned, neither the ashing nor the digestion procedure showed any great advantage. However the following advantages and disadvantages were noted by the different laboratories.

The ashing procedure is relatively quick and uses a minimum of reagents but care must be taken not to blow away any of the ash and not to let the temperature exceed 400° C.

The digestion procedure eliminates the danger of loss of ash and the temperature is always below the danger point. On the other hand it may require a longer period of time, particularly with the animal fibers, and much larger quantities of reagents are used. In the case of the digestion process, some laboratories reported a correction for the reagents of as high as 75 λ of copper, which is somewhat excessive when only 100 λ of copper are being determined. There is also, naturally, a much larger concentration of salts in the solution from the digestion procedure. However, if the sample to be analyzed is 1 gram or less most of the objections to the digestion procedure are removed.

As far as methods of analysis were concerned, all the laboratories were agreed on eliminating from consideration the potassium ethyl xanthate method. In the first place the yellow color was rather difficult to match accurately, but more important still was the rapid change in color of both the unknown and the standards.

The colorimetric ferrocyanide method was preferred by several laboratories, particularly from the standpoint of accepting or rejecting fabrics on the basis of a definite tolerance limit for copper. On the other hand, the color comparison between the unknown and the standards is often rendered quite difficult by a cloudiness which develops in the unknown solution and obscures the color. Unless the samples to be analyzed are 5 grams or over, the accuracy of this method is seriously questioned.

The volumetric method utilizes a technique with which all analytical chemists are very familiar, and it does away with any difficulties of color comparison due to the cloudiness of the solution. On the other hand 1 c. c. of N/1,000 sodium thiosulfate is equivalent to 63.6 λ of copper, so that only 1.57 c. c. are required to titrate 100 λ of copper. This practically necessitates the use of a micro-burette. Also the N/1,000 sodium thiosulfate is quite unstable and must be made up fresh at least every day. This method is not recommended for quantities of copper under 100 λ.

In conclusion we might state that the two last mentioned methods—namely, colorimetric with potassium ferrocyanide and volumetric with sodium thiosulfate—are the only ones which are at present being extensively used by textile chemists: the colorimetric method being preferred if it is only necessary to determine whether a sample is within a definite tolerance limit for copper, and the volumetric method being principally used if it is desired to determine the percentage of Cu in an unknown sample. The accuracy of both of these methods is somewhat questionable if the amount of Cu to be determined is less than 100 λ.

In the opinion of the writer there are two methods which offer definite possibilities of being adapted to the needs of the textile chemist and the manufacturer of rubberized fabrics:—namely, the colorimetric method using sodium diethyldithiocarbamate and the volumetric method using the nitroso-chromotropic reagent. It is to be hoped that further investigations in the near future will develop standard procedures by which one or both of these methods can be applied to the determination of copper in textiles even in amounts as small as 10 λ, thereby making it possible to analyze a fabric sample of 1 gram or less.

. . . At the conclusion of his paper, Dr. Scott made the following comment:

I hope that our own Association will be able to devote a little more time on the part of one of our Research Associates to investigating the value of these last two methods. I think that would be very much worthwhile.

Thank you. (Applause).

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Chairman Cady: Is there any discussion of this paper? (There was none.)

The final paper on this morning's program will be an illustrated talk on, "The Rayon Industry—A Quarter Century of Progress," by Dr. Harold D. W. Smith of A. M. Tenny Associates. (Applause).

The Rayon Industry

A Quarter Century of Progress

By **HAROLD DE WITT SMITH,**
Ch.E., Ph.D., F.T.I.

Textile Technologist, A. M. Tenney Associates, Inc.

THE purpose of this great World's Fair in Chicago is to offer visible evidence of a century of progress with particular emphasis on science and industry. A century ago, the rayon industry had not been born, nor scarcely conceived except in the casual but prophetic suggestions which we find in the writings of Bacon & Reamur.

Twenty-five years ago all four of the present commercial processes had been discovered and three of these were in actual operation abroad. "Artificial Silk" or "Wood Fiber Silk" was on the market in braids and trimmings and weird looking knitted mufflers and neckties and in socks which showed a disconcerting tendency to disintegrate in the wash tub. My own connection with the "man made" fiber covers only slightly more than a decade but in this last tenth of the century whose progress is being chronicled here at the Fair, I have witnessed first hand one of the most astounding fruitions of scientific and industrial research.

Let me try to picture for you the progress which I have seen, not by recounting a mass of details, but by touching briefly and graphically on a few of the significant highlights.

What do we mean by progress? In any branch of the textile industry (except for a few mechanical uses in which fibers are judged by engineering standards and measurements), and most particularly in rayon which like silk is preeminently a woman's fiber, the ultimate measure of progress is the *woman's* verdict on the materials which we textile men create.

It is in order to win her approbation that we must develop the mechanical, physical and chemical properties of our synthetic fibers to the point where the fabrics which attract her are practicable to make and practicable as well as ornamental in use. Therefore, the technical progress of which we are so proud is in this sense an *effect* rather than a cause. And the rather amazing statistics of production which are most often quoted to show the progress which the rayon industry has made are but a drab yardstick by which to measure the *consumers* acceptance of the technical progress of the industry.

Let us first have done with the *quantitative* evidence of statistics. Twenty-five years ago there was no rayon industry in the United States and the world production was about one-twelfth of the amount which will be produced in the United States in 1933 and only one thirty-seventh that of the present world production.

*Presented at Saturday morning session, Annual Convention, Chicago, September 9.

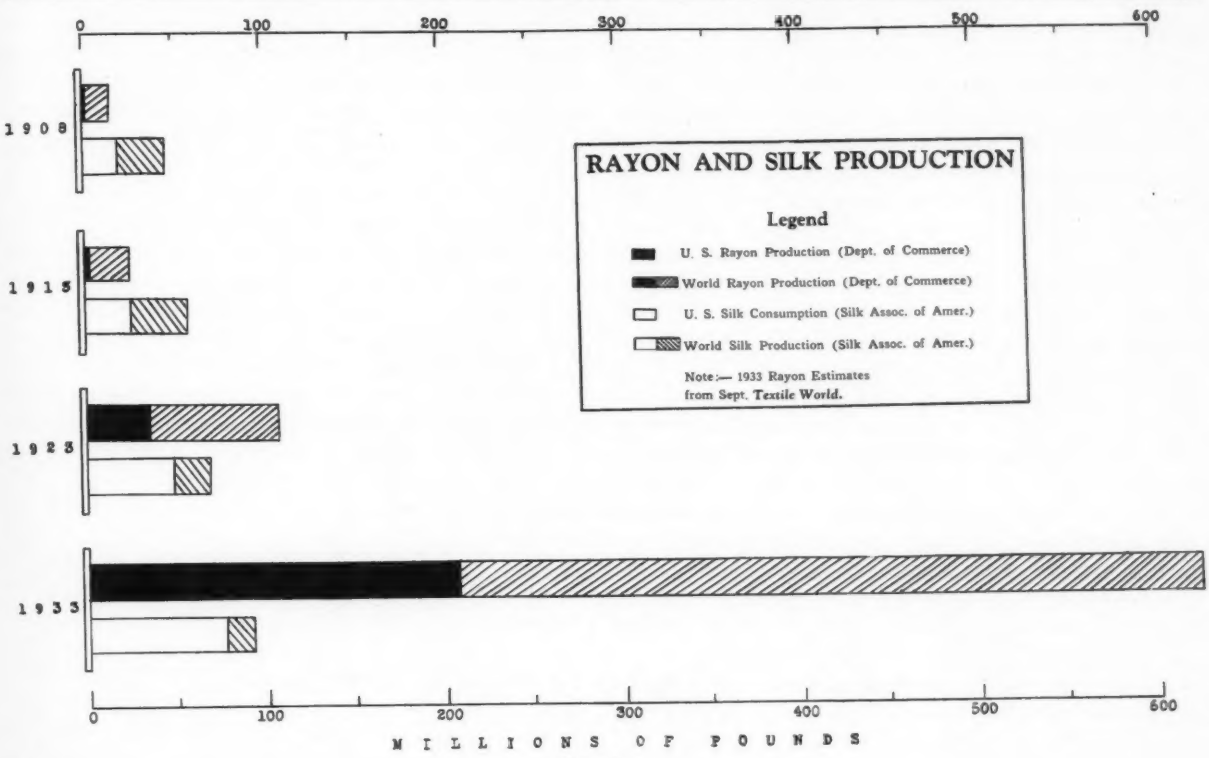


Figure 1.

During the last ten years, the amount produced in the United States has ranged between one-quarter and one-third of the world production. The estimate made by *Textile World* for 1933 show a world production six hundred and twenty-six million pounds which is almost seven times the production of natural silk. In this connection, it is interesting to note that Japan, which is the world's principal source of natural silk, has become rayon conscious to such an extent during the past decade that from a mere eight hundred thousand pounds in 1923 her production of rayon has risen eighty-eight fold to seventy million pounds in 1932, which puts her in second place among producing countries.

We may dismiss production statistics with a glance at Fig. 1 which substitutes a simple graph for a mass of figures.

This quantitative picture is impressive but it tells us nothing about which kind of progress we are making. What sort of yarn is being made as the years go by, and into what sort of fabrics and garments is it going?

For *qualitative* evidence we must turn first to the yarn producers for technical progress, and then to the fabric producers for artistic progress. We must include here the weavers who have developed the technique of processing the yarn and of producing finer and finer textures and also the dyers, printers and finishers who have developed new beauties of color, of texture and of design.

From a *qualitative* standpoint, we have very little data for 1908. There *was no* production in this country and the imports, which totaled not quite one million pounds, were confined principally to braids and trimmings and seamless hosiery.

Even twenty years ago there were only two or three yarn numbers manufactured in the United States, whereas, today, counting dull and bright varieties, there are 286 different yarns available to the weaving and knitting technicians.

A glance at the evolution of *filament structure* offers very significant evidence of the progress made toward more silk-like yarns.

Twenty-five years ago, the imported 150 denier viscose yarn was composed of only *fourteen* filaments. In other words the denier of the single filament was 10.7. This compares to the denier of a single filament of Japanese silk which after boiling off is about 1.3.

Keeping this 1.3 denier in mind as a yardstick for measuring fineness of filaments, we find that twenty years ago the filament count of 150 denier yarn had risen to 16 or 9.4 denier per filament. Ten years ago, the bulk of 150 denier yarn produced was made with 18 filaments of 8.33 denier and the finest filament structure offered in viscose yarn was of 6.25 denier. At the present time the ordinary viscose filament count for 150 denier is forty which means that the denier of a

single filament is 3.75 and almost every Viscose manufacturer is offering 150 denier yarns containing sixty and in some cases ninety filaments or a filament denier of 2.5 and 1.7 respectively. The average acetate filament structure of the present day is about three denier and finer filament yarns can be had of 1.88 denier. The cuprammonium type, whose commercial production has been chiefly limited to one company, has always had a filament denier comparable to that of silk, namely 112 filaments for 150 or 1.35 denier. Figure 2 will perhaps help to visualize the importance of this evolution toward finer filaments. In this figure the filaments are diagrammatically represented by *cylindrical* rather than *actual* cross section. In 1908 such a filament had a diameter of 40.4 microns. Since the rigidity of a cylindrical rod increases as the *cube* of the diameter, the rigidity of a filament of such size is thirty-nine times that of a filament of the same material the size of a silk filament. The successive steps toward the present day filaments of viscose whose diameters approach that of the silk filament with correspondingly lowered rigidity is indicated in the figure. Of course, other factors such as the actual shape of the filament and the material of which they are composed affect the wiriness or rigidity of the yarn. One of the advantages of acetate yarn is its inherent softer nature. Nevertheless this evolution toward finer filaments is strongly indicative of the improvement in draping quality and softness of hand which has occurred and it has also much to do with improved dyeing qualities, strength and other properties.

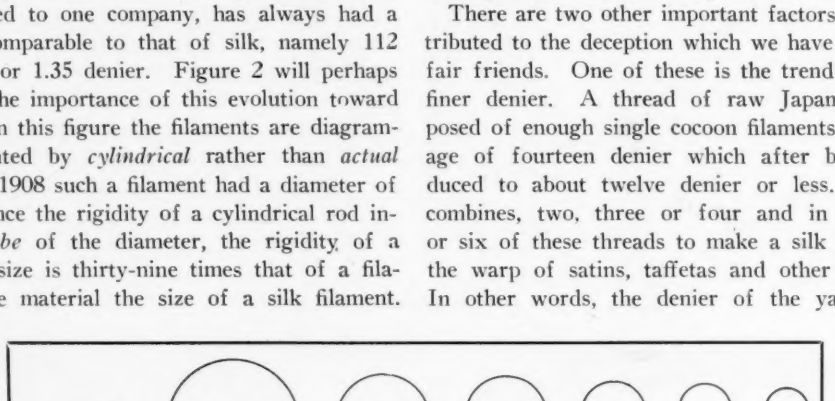


Figure 2

The significance of this steady increase in the fineness of the individual filaments is undoubtedly greater than that of any other single factor in gaining the acceptance of the "man-made" fibers by women. I venture to say that each of you who has a wife or a friend of the gentler sex has been told that rayon is "too shiny," or "too stiff, harsh and wiry." All of this was true some years ago, but while the ladies have continued to hold this picture of the synthetic yarns in mind, we have been slyly developing these finer filament structures which have resulted in a softness of the yarn and a texture of the fabric much nearer to that of silk. So near to it, in fact, that many of the women who assure us that they do not like our products, have undoubtedly been wearing them in fabrics which they have purchased because of their quality rather than of price alone.

There are two other important factors which have contributed to the deception which we have practiced on our fair friends. One of these is the trend toward yarns of finer denier. A thread of raw Japanese silk is composed of enough single cocoon filaments to give an average of fourteen denier which after boiling off is reduced to about twelve denier or less. The throwster combines, two, three or four and in some cases five or six of these threads to make a silk yarn suitable for the warp of satins, taffetas and other fine broad silks. In other words, the denier of the yarns in such silk fabrics range from about twenty-four to seventy-two. Contrast this with 150 denier rayon which is equivalent to twelve thread silk. Except for a few hardy pioneers among the mills, even this number was used principally as filling until some six or seven years ago, because of the difficulties encountered in weaving rayon warps. It is really only within the last three or four years that the skill of the weaving technicians and that of the rayon producers have combined to provide on a broad commercial scale fine sizes comparable to those of silk in the silk constructions and textures of the present-day rayon fabrics.

As evidence of the trend toward finer deniers the following figures are interesting:

In 1908 and also in 1913 all of the imports and production of the Viscose Company was of 150 denier or coarser. They estimate that in 1908 about 80% of the total was 150 denier and in 1913 about 90%. By 1923 viscose of 120 denier was being made and nitrate and acetate yarns as fine as forty-five or fifty denier but the quantity of these was relatively small.

In 1929, according to figures compiled by the

"Textile Organon," 11% of the total domestic production was finer than 150 denier. By 1931 this figure had increased to 16% and in 1932 to 25%. The deniers offered at present range from 15 denier on the fine side to 1,200 denier on the coarse.

The other important factor is the development of *dull* luster yarns of various types. These have reduced to a more conservative luster the shine or gloss which, although apparently an asset in "ancient" times, that is fifteen or twenty years ago, had gradually become distasteful to the fair consumer. In fact, the pendulum of the mode swung on toward a dullness exceeding by far that of natural silk, and the synthetic fibers because of their adaptability to such style trends followed through to chalky flat white effects that are scarcely obtainable in silk.

With regard to *strength,* there is little or no actual published data available but undoubtedly a continuous gradual improvement in the tenacity and toughness and a consequent reduction in the brittleness of the filament has occurred. Part of this has been an inevitable accompaniment of the increasing fineness of the filament because the surface or skin of each filament is always better oriented and therefore stronger than the interior and as we have increased the fineness, we have, of course, greatly increased the ratio of surface to volume.

Perhaps the most graphic commentary on the improvement in strength during the last twenty-five years is found in the *only* technical reference to "artificial silk," as it was then called, which I could discover in the "Textile World Record" for the year 1908. Among the questions to the Technical Editor was one from a manufacturer of braids for trimming dresses who stated that in damp weather the artificial silk which he was using was so greatly affected that it was impossible to run his braiding machines and who begged for information as to how to overcome this difficulty. The reply was brief and to the point, namely "it is the custom among manufacturers of braids of this class to stop their machinery whenever the weather is very damp. There has never been, up until this time, any remedy offered."

Since that time we have learned to produce rayon filaments which are stronger than the cotton fibers from which they are made; too strong, in fact, to be good, for we have learned that strength, like every other property which we have striven to improve, is important in its relation to the others rather than by itself alone. In other words, the success which rayon has achieved to date is due to the attainment of coordination or balance of all of the properties which contribute to the final quality.

On the general improvement in uniformity of dye absorption in recent years, I need hardly dwell before you chemists and colorists who have yourselves observed this change in the goods which pass through your hands year after year. But I would like to emphasize here once more the part which the dyer and finisher of rayon fabrics has played in obtaining the beautiful colors, finishes and effects on present day materials.

Many of you are as old or older than I am in this young industry and have seen for yourselves the changes which I have tried thus briefly to chronicle. You know, also, as well as I do, that the end is not yet; that ten and twenty years from now we shall look back upon our present synthetic fibers with faint amusement as we compare them to the newest products of that time. As a graphic summary, however, which speaks louder than any words of mine of the progress which the industry has made to date, as measured by the woman's subtle judgment, I have brought a few swatches of "ancient" and modern fabrics for your examination.

Discussion

Chairman Cady: Is there any discussion of this paper? I suggest that after adjournment of this session those who are interested in seeing these fabrics come forward and I am sure Dr. Smith will be glad to explain them in more detail.

At this time we will adjourn until after lunch and we will commence the afternoon session as soon after two o'clock as possible. The first speaker will be Dr. Wayne A. Sisson of the University of Illinois.

. . . The meeting thereupon adjourned at one-fifteen o'clock . . .

UNEMPLOYMENT REGISTER

Members of this association, who are without employment, should forward to the secretary a statement as to their qualifications and experience. This information will subsequently appear on these pages with an identifying key number. Employers are also requested to file with the secretary any vacancies which may occur in their businesses.—A. Newton Graves, Secretary, care Franklin Process Company, Providence, R. I.

Key: A—Laboratory and research; B—Dyeing or printing; C—Finishing; D—Sales; E—Instruction; F—Executive

NOTE. Persons who have obtained employment should notify the secretary at once. Prospective employers may file vacancies either with the secretary or the AMERICAN DYESTUFF REPORTER.

A-2

Education—B.T.C., Lowell Textile Institute, 1932; also course in shorthand and typing.

Experience—None to date, interested in quantitative analysis, experimental dyeing, color matching, dye testing and dye identification. Ability to learn quickly and will go anywhere. 23 years old, single.

A-B-1

Experience—Over twenty-years' experience in the laboratories of various chemical manufacturers, four months' experience as second hand in dye house, familiar with all types of dyes and fibers. Thirty-nine years old. Will supply references.

A-B-2

Education—Lowell Evening Textile School, 1917.

Experience—Five years' experience in hosiery dyeing, full fashioned hosiery of silk and cotton and silk, also men's hosiery containing rayon, wool, cotton and celanese. Several years experience in laboratory work. Has also had experience in jig-dyeing of cotton piece goods, slub and top dyeing.

A-B-3

Experience—Wide experience in several of the best print works in New England as plant chemist and application chemist. Has had practical experience in the supervision of several departments and in the development of processes. References furnished.

A-B-C-1

Education—Graduate Industrial Chemical Engineer, Pratt Institute—1911.

Experience—One year as plant control chemist, 4 years as laboratory chemist with gas company, 13 years as plant chemist with bleaching, dyeing and finishing plant. (Rayon and cotton piece goods). Formulated processes and put them into plant operation, 1 year as chemist and demonstrator for dyestuff company, 1 year as superintendent colorist with print works printing linen and rayon, 1 year as director of testing laboratory for national dry goods concern.

A-B-E-1

Education—B.S. in chemistry at C.C.N.Y. Working now on M.A. at Columbia.

Experience—7 years as dye tester and color matcher for dye manufacturer; 6 months skein silk and rayon dyer; 4 years textile chemist and dyer for cotton finishing concern; 2 months boss bleacher and 4 years as teacher of organic chemistry and dyeing. Age 38, single.

A-E-1

Education—Ph.D., Univ. of Illinois, studied chemical engineering at Columbia, licensed chemical engineer, New Jersey.

Experience—10 years' experience teaching in colleges, subjects included chemical and textile subjects. Has had broad experience in industrial work including experience in testing materials, research and consulting. Will go anywhere; references.

A-E-2

Education—B.T.C., Lowell Textile Institute, 1933.

Experience—Assistant instructor in chemistry, Lowell Textile Institute. Member of scholastic honorary fraternity.

B-1

Education—Educated and trained in chemistry and calico printing in Glasgow, Scotland.

Experience—Has been with large print works as colorist for 13 years producing first class work on cotton, rayon, wool and silk fabrics. References.

B-2

Experience—Boss dyer with one firm for 18 years, divisional supt. at print works for 1 year, in charge of processing and dyeing of cottons and rayons at large bleachery for 6 years. Prefers bleachery in East but will go anywhere. References.

B-3

Experience—Foreman piece dyer for the past 21 years. Can dye the following; celanese, rayon, cotton backs, wool, silk and mixed fabrics. Has knowledge of finishing and laboratory work.

B-6

Education—Graduate R. I. School of Design.

Experience—5 years in bleachery, 7½ years as assistant dyer, 2 years as boss dyer and 3 years as superintendent of dyeing and finishing. Married, 33 years old. References.

B-7

Experience—4 years as assistant to manager in ribbon mill; 2 years as boss dyer on veils, chiffons and silk skeins; 5 years as boss dyer on ribbons and skeins (cotton and silk); 7 years as boss dyer on spun silk (pure dye and fast) resisted colors; last 12 years as hosiery dyer on silk and cotton, silk-cotton, rayon or wool. 50 years old, married. References.

B-8

Education—Chemistry and Dyeing Department, Philadelphia Textile School.

Experience—Eleven years experience as chemist and boss dyer on the following: woolen, worsted and cotton yarn; woolen, worsted and union piece dyes on both ladies and men's wear; wool, cotton, shoddy and mohair raw stock. Age 33, married, will go anywhere. References.

B-9

Experience—Hosiery dyer with fifteen years' experience on high grade silk hosiery; familiar with all types of machines. Age 43, married. Will go anywhere. References furnished.

F-1

Education—Textile Institute, Alexander Hamilton Inst., Extension work at Brown Univ., working knowledge of German.

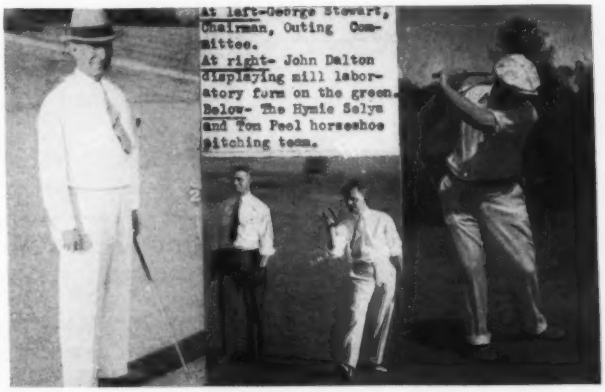
Experience—Has had executive departmental work with all types of textiles manufacturers. Acquainted with all types of textile machinery, dyestuff application, laboratory practice, cost methods, and checking methods and processes. References.

OUTING, NORTHERN NEW ENGLAND SECTION

THE Eleventh Annual Outing of the Northern New England Section was held at the Vesper Country Club, on Friday, June 16, with Roland Derby and George Stewart, both members of this club, acting as hosts, and as members of the arrangement committee.

The staff at Vesper made very delightful arrangements at the Clubhouse for the visiting members. It was a pleasure to visit Vesper, as Vesper was the scene of the first outing held by the organization some ten years previous.

After a delightful day with keen rivalry among the tennis players, and the good fellowship among the golf players, a banquet was held in the evening, attended by approximately one hundred members and guests. No speaking program was arranged, but with the music engaged by the entertainment committee, and the various groups of good fellows discussing everything from golf balls in the rough to the pH value of a dying moth, a delightful evening was spent.


At left-George Stewart, Chairman, Outing Committee.
At right- John Dalton displaying mill laboratory form on the green.
Below- The Hymie Selya and Tom Peel horseshoe pitching team.

Chairman Leverett Putnam presided, and a vote of thanks was given to the outing committee. Reports of the Chairman, Secretary and Treasurer were read and accepted, and the results of the ballots for the officers for the ensuing year was announced as follows:

Chairman .....Leverett Putnam
Secretary.....Clarence Nutting
Treasurer .....John H. Grady
Sectional Committee for two years:
Edward Weber and Braman Proctor.

(Claxton Munro and Stephen Bastow remain on the Sectional Committee, having been elected last year for two years.)

Representative to National Council—Roland E. Derby.
Representative to National Nominating Committee—Henry D. Grimes.

The prize winners for golf were as follows:

Members

Best Gross—Won by M. P. Graham.
Best Net—tie, W. Zillessen, J. A. Bryant, Sr., J. H. Grady.
Drawing for net prize won by W. Zillessen.

Guests

Best Gross—Won by C. R. St. Clair.
Best Net—Won by W. A. Ritz.
Long Drive—Won by J. A. Bryant, Jr.

TENNIS

Singles won by Chas. H. A. Schmitt.
Doubles won by Hyman P. Selya, Norman Hamel.

Lucky Number

First prize—Harold W. Leitch.

Second prize—A. M. Reid.

Due to efficient conduct of the outing by the committee under Chairman Stewart the affair was entirely self-supporting, no money was required from the sectional treasury.

Respectfully submitted,

CLARENCE L. NUTTING,
Secretary

STANDARD SILK SAMPLES

There have been prepared, under the direction of the Sub-Committee on Fastness of Dyed Silk, standard dyed silk fabrics to represent four classes of fastness to washing—namely, Class I, Class II, Class III and Class IV. These standards have been carefully dyed with the dyestuffs and according to the dyeing methods recommended in the 1931 Year Book.

The A. A. T. C. C. is prepared to furnish sets of these washing standards for a nominal charge which will cover the cost of preparation. It will be possible to accurately grade the fastness by comparing it with the standards after subjection to the standard washing tests approved by this Association. All inquiries concerning these Silk Washing Standards may be addressed to the Chairman of the Research Committee.

Louis A. Olney,
Lowell Textile Institute,
Lowell, Massachusetts.

AMERICAN
DYESTUFF
REPORTER

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AN ARM OF THE GOVERNMENT

THE title of this editorial is taken from the characterization given the chemical industry by W. B. Bell, president of the American Cyanamid Company and also president of the revived Chemical Alliance, Incorporated, at the recent code hearing in Washington on the chemical industry. At the hearing Mr. Bell gave a general description of the chemical manufacturing industry and the gist of his remarks along this line brings out the following facts:

He explained that the Chemical Alliance was formed during the war but, since the Armistice, has been inactive. With the advent of the NRA and its resultant codes, the Alliance was revived as a non-profit organization for the purpose of stimulating co-operation between the government and the entire chemical industry. In order to accomplish this purpose, every branch of the chemical industry was given representation on the Board of the Alliance and as a result the 238 members who are so far members of the Chemical Alliance employing between 50 and 60 thousand persons are represented. The total membership will probably be close to 500 firms who will employ in the neighborhood of 75,000 persons.

Mr. Bell further brought out that inasmuch as the chemical industry had developed under the help of the government during the war and, emphasizing the value of chemistry to science and to all industry, he stated that the chemical industry should be considered practically a part of the government.

The "merit" clause in the chemical code which caused considerable discussion in an entirely different code of another industry was the subject of some of Mr. Bell's remarks. Commenting editorially on this the *Oil, Paint and Drug Reporter* states:

"Talking with spirit apropos the 'merit' clause, which has crept into one other major code and which has been the subject of assault by the organized labor interests, Mr. Bell felt that it had a distinct place in the chemical code, above all others, because of the nature of the work and the type of employees engaged in it. Employees even in minor positions, declared Mr. Bell, frequently are making important discoveries and suggestions and not to reward such employees for their discoveries, not to base promotions on merit, will mean the end of scientific advancement, in his opinion. We cannot quite agree that a code which is designed only to be operative for a restricted time would have that cataclysmic effect, but we do agree that it is ridiculous to regulate laboratory workers and scientists—acknowledged or budding—on a strict eight-hour or any other hourly basis. We laugh, or shudder, when we think of what would have resulted had Edison put himself on an NRA code schedule at Menlo Park.

"Many of the points in the chemical code are designed to give the required elasticity or flexibility to the industry and its technical workers.

"With regard to the factory employees, the situation, said Dr. Bell, is also different; and because unemployment in the chemical industry is less than in other basic industries and further because of the emergency needs for continuous or unusual operations, he spoke strongly in favor of the wage and hour clauses provided in the code."

We also agree that it is quite absurd to put a scientist or technical worker on a basis of hourly research, and the worker himself would not desire that. Scientific research to a true scientist is not considered as "labor" but as an accomplishment when completed. To put him under a code restricting the hours of work would stifle ambition and initiative. The case of a chemist differs considerably from that of the average factory employee.

Mr. Bell further declared that it was not necessary for the chemical industry to cut hours and increase wages in order to do its share in restoring pre-depression purchasing power. He used various statistical figures to support this contention. He also brought out the danger in attempting to distribute the unemployed among the various industries as has been suggested by some, declaring that it is impossible to permanently raise the economic condition of the country by raising wages and then raising tariffs to counteract increased costs. He then stated that by impairing the efficiency of the chemical industry by higher labor costs, all industry, and the government itself, would suffer, because chemistry is the most basic of industries.

Whatever the personal views of the reader may be, it must be recognized, as stated by Mr. Bell, that the chemical industry is basic and should be considered now and in the future as an "arm of the government."

Wetting Agents in Textile Processing

III. INFLUENCE OF WETTING AGENTS UPON THE ADSORPTION OF ACID AND BASIC DYES BY WOOL

By **HARVEY A. NEVILLE and CHARLES A. JEANSON, III**
Department of Chemistry, Lehigh University, Bethlehem, Pa.

ONE of the uses for which wetting agents of the new type are recommended is that of leveling or retarding the adsorption of the coloring matter in the dye bath. It was, therefore, considered of interest to determine quantitatively the actual influence of a wetting agent upon the amounts of typical dyes adsorbed by a textile material such as wool.

The wool used for these experiments was a fine, white worsted knitting yarn. It was maintained at constant humidity, and one meter (about 0.5 gram) was accurately weighed for each test. Methylene blue was used as the basic dye at a concentration of 0.2 per cent; the acid dye was Orange II, and the concentration of the dye solution was 0.1 per cent in this case. Series of experiments with each dye were also performed in which Gardinol was added to the extent of 0.1 per cent and 0.5 per cent of the weight of the solution. The samples of wool were boiled for two minutes in 50-c.c. portions of the dye solutions which were made acid or alkaline with sulfuric acid or sodium carbonate respectively. Each sample of wool was transferred from the dye bath to distilled water and boiled for two minutes. It was then placed in an Erlenmeyer flask, covered with distilled water, and the air was displaced from the flask by carbon dioxide. The contents of the flask were heated to boiling and the dye on the wool was titrated with titanous chloride, using a slight excess of this reagent. The boiling was continued until the color was completely discharged and the excess of titanous chloride was then titrated with a solution of the same dye which served as its own indicator. A steady stream of carbon dioxide was passed through the flask throughout the titration. The pH values of the dye baths were obtained, after removing the wool, by means of a quinhydrone electrode.

The results for the acid dye are shown in Fig. 5 and for the basic dye in Fig. 6. The percentages shown are based upon the weight of the wool. Quantitative data for the adsorption of acid and basic dyes have been published by Briggs and Bull[9] who also studied the effect of the addition of salts to the dye bath. The results represented by curve I for each dye are consistent with the data of Briggs and Bull and with Bancroft's generalizations for the adsorption of dyes[10]. With regard to the influence of the wetting agent or leveler, this can best be interpreted from a consideration of the nature of the surface-active ion in relation to the electrical condition of the wool (Cf. Part II, Fig. 4). As shown in Fig. 3 (Part II), Gardinol is strongly adsorbed by wool in acid solutions and, since its active ion is negative, this interferes with and decreases the adsorption of an acid dye in which the surface-active ion is also negative. In the case of the basic dye in which the active ion is positive,

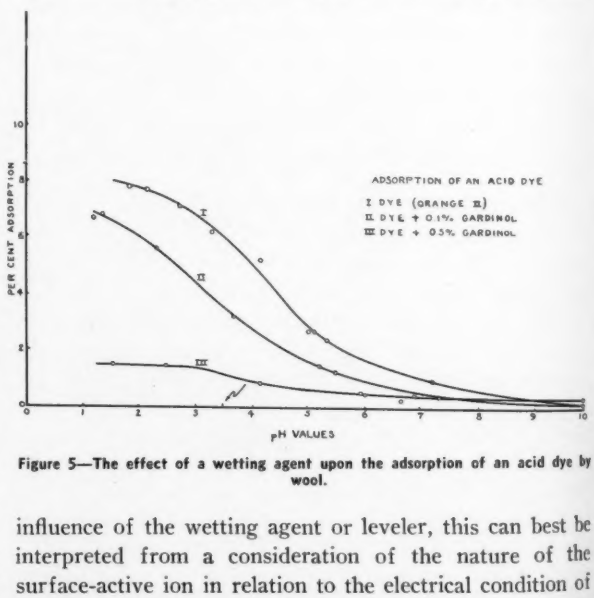

Figure 5—The effect of a wetting agent upon the adsorption of an acid dye by wool.

(Continued on page 621)

SHRINKAGE OF COTTON AND LINEN WOVEN MATERIALS

(Continued from page 592)

(j) The Effect of Pressing

The question is often asked "What rates of contraction or shrinkage occur when fabrics are laundered under slightly different conditions in a number of plants? Will the same contractions develop or will they be entirely different?"

In order to answer this question, samples of cloth taken from one bolt of material were prepared, measured for length and width, and were then sent to ten different laundries to be washed and finished three times with the fast colored lots. The washing temperatures used ranged for this class of work between 120° and 140° F. In one or two cases a washing temperature as low as 100° F was used. The number of suds used ranged between 3 and 4, depending upon the plants. Four to five rinses were used. In other words, the washing formulas varied slightly according to local conditions of soil. The fabric used, however, was the same in all cases, taken as it was from one piece of cloth.

The following results were obtained after 3 washings and finishings, the original warp dimension equalling 30 inches and the filling 29 inches.

Table XVII
Commercial Laundry Unit

	1	2	3	4	5	6	7	8	9	10
Lgth.	30.0"	30.0"	29.6"	30.3"	29.8"	30.0"	30.0"	30.0"	29.8"	29.9"
Wdth.	29.3"	29.3"	29.4"	29.2"	29.5"	29.3"	29.7"	29.4"	29.4"	29.3"

It will be noted, compared with an original length measurement of 30 inches, there were five instances wherein the warp dimension proved to be the same after three launderings as it was before. In three instances the contraction equalled only 0.1 to 0.2 of an inch. In one case the contraction equalled 0.4 of an inch, while in another a gain of 0.3 of an inch developed. In other words, in spite of variations occurring in the method of washing used, there was only one case where any particular contraction occurred, i.e., Plant Number 3. In all cases the filling showed a slight gain, indicating that a slight stretching had occurred.

As a matter of fact, the slight variations shown in the table in all probability were due not to differences in behavior within a washer cylinder itself, but instead to variations in tension that were applied by press operators when the pieces were smoothed out on a buck prior to applying the steam-heated head to the cloth surface. In other words, if one operator of a press fails to eliminate all wrinkles or to otherwise stretch the fabric while another girl does take the precaution to smooth the fabric surface, slight but not always serious discrepancies are almost certain to develop. Variations in the amount of tension that is applied in order to smooth out wrinkled surfaces often account for slight differences in shrinkage tests such as are shown in Table XVII.

A second series of samples were laundered three times by nine different plants and the following results were obtained. Again it will be noted that slight discrepancies have occurred, but that in general the results obtained by all nine plants are largely comparable. The length originally equalled 30 inches and the width 37 inches.

Table XVIII
Commercial Laundry Unit

	1	2	3	4	5	6	7	8	9
Lgth.	29.2"	28.9"	29.1"	29.6"	28.8"	29.1"	29.3"	28.8"	29.1"
Wdth.	37.3"	36.9"	36.8"	36.8"	37.5"	37.1"	37.3"	37.1"	37.2"

The importance of tension upon pressing is particularly pertinent to a commercial laundry shirt unit. Certain shirt manufacturers have frequently pointed out the tendency for sewing threads in neckband shirts to bunch into V-shaped groups, thereby causing the sewed fabric to contract. This bunching undoubtedly is due to the difference in shrinkage or contraction of the sewing thread compared with the cloth used in the neckband. When such neckbands are hand ironed, there is a natural tendency for the bunched yarns to be stretched. When the neckbands are pressed on a shirt unit, however, unless the press operator jerks each neckband or collar carefully so as to stretch the bunched threads, a slight contraction is likely to take place. If the amount of tension applied varies with the occasion or with the operator, again differences in length will occur with repeated laundering. The same general fact is largely true of a sleever. If shirt sleeves are pulled over a pair of sleevers with greater tension during one trip through a plant compared with another, there sometimes will be a great tendency for the sleeves to be stretched filling or crosswise, a fact which causes their length to be somewhat shortened.

The necessity for stretching washsuits at the time of pressing also is advised. This recommendation is made with the realization that small amounts of shrinkage can be overcome with the adoption of such a policy. It is clearly recognized that excessive amounts of shrinkage cannot be controlled by such methods. Excessive rates of shrinkage—as is realized by all laundryowners and many washsuit manufacturers and retailers—require the use of a recognized preshrinking process prior to tailoring.

The variations in tension that are applied by an operator to articles being pressed frequently accounts for slight discrepancies as far as the shrinkage of cotton and linen fabrics are concerned.

(k) The Effect of Home vs. Commercial Laundry Washing Methods Upon Shrinkage

Previous data should answer all questions with regard to this point. If a fabric has any tendency to contract or shrink, shrinkage will occur during the first laundering, regardless of the washing process used. During 1926 the Procter and Gamble Laboratories published comparative home and commercial laundry shrinkage data, showing that after twenty repeated launderings, no significant shrinkage differences existed. These data are shown in Table XIX.

Table XIX

	Home Laundering Method			Commercial Laundering Method		
	Warp Measurement			Warp Measurement		
	Original	After 20 Washings	Shrinkage in percentage	Original	After 20 Washings	Shrinkage in percentage
1	33.5"	29.5"	11.9%	34.0"	30.0"	11.7%
2	33.5	30.5	8.9	34.0	32.0	5.9
3	33.0	30.0	9.0	33.0	30.5	7.5
4	33.2	29.5	11.2	33.5	31.0	7.4
5	34.0	30.2	11.0	33.5	30.0	10.4
6	33.5	31.5	5.9	33.5	32.5	3.0

(1) *Summary of Shrinkage Data*

The following conclusions have been drawn relative to the shrinkage of cotton and linen woven materials.

1. The amount of shrinkage obtained varies with the material, depending upon the treatment that a given wash fabric receives during mill processing.

2. There must be moisture present for shrinkage to occur.

3. The amount of shrinkage obtained in a soaking process is less than that obtained during actual laundering. This fact is particularly true of heavily starched materials.

4. An increase in washing temperature does not have a marked influence upon the rates of shrinkage.

5. The presence or absence of washing supplies has little, if any, effect upon shrinkage. Supplies chiefly assist in wetting out the material as far as shrinkage is concerned.

6. Shrinkage occurs regardless of the washing process used, unless the fabrics being laundered have been properly preshrunk prior to tailoring.

7. The greatest amount of contraction occurs during the first laundering.

8. Unsized materials shrink very rapidly.

9. The warp shrinkage generally is in excess of the filling shrinkage.

10. Pressing with a hand iron tends to stretch fabrics slightly, but will never overcome excessive warp or filling shrinkages.

11. Slight variations in size after different launderings often may be traced to variations in tension as applied by operators at the time of pressing. Serious shrinkages cannot be traced to such a source, however.

12. It is advisable to stretch washsuits, shirt collars, neckbands, etc., at the time of pressing.

13. Shrinkage is the same, within the limits of experimental error, whether cotton and linen fabrics are washed under commercial laundry or home conditions.

14. A gain in yardage during mill finishings means shrinkage during laundering.

15. There is need for the use of properly preshrunk cotton and linen wash fabrics in the manufacture of any article that is tailored to fit.

16. Unless a wash fabric has been preshrunk by some satisfactory method, there is nothing that can be done during laundering to prevent contraction from occurring.

Present Trends in Preshrinking

The commercial laundry industry is gratified to note the manner in which responsible textile houses have adopted preshrinking methods. It is estimated that during 1933 between two and three hundred million yards of cloth will be preshrunk prior to being offered to the public. This is encouraging when it is recalled that but a few years ago many textile manufacturers and converters refused to adopt suitable methods, because the surface luster was destroyed or the pattern was altered slightly during the process. The policy of tailoring garments from fabrics that obviously have been stretched during the finishing process and then expecting either the consumer or the laundry to get along as best they may has been definitely discarded by many concerns as basically unsound.

Choice of Proper Method of Preshrinking

Obviously the first concern of a manufacturer, who is interested in preshrinking, should be the efficiency of the shrinking unit that he adopts. Most authorities agree that there are a number of entirely satisfactory shrinking methods on the market that materially aid in eliminating consumer complaints. On the other hand, there are other preshrinking methods, such as some of the older water-shrinking processes, that do not show the same efficiency. A manufacturer can very quickly check the efficiency of any preshrinking unit in which he is interested by having a sufficient number of samples handled by the unit washed under commercial laundry conditions at the American Institute of Laundering. One hardly needs to be a prophet to predict that when improperly preshrunk garments are sold to consumers, their reaction to the retailer that sells such merchandise as "preshrunk" is not a pleasant one.

The Necessity for Using Care When Tailoring

In addition to choosing wash fabrics that have been correctly preshrunk, a cutter finds it necessary to pay considerable attention to the tailoring methods that are used. It should not be expected that a preshrunk fabric is converted into a rigid material that cannot be stretched with ordinary handling. Recognizing that improper tailoring methods may stretch preshrunk goods, Cluett, Peabody and Company, for example, give the following recommendations to garment manufacturers who use sanforized materials:

"Fabrics that are completely shrunk are more elastic than partially shrunk or unshrunk fabrics, and therefore have a tendency to elongate when subjected to tension.

"To avoid distortion of a sanforized fabric in manufacturing operations, the following suggestions should be carefully observed:

"1. Make sure that cutting patterns are the proper dimensions to produce a garment of the exact finished size desired.

"2. Do not pull the material while spreading it on the cutting board.

"3. Do not dampen or sponge material before cutting or after placing it on cutting board.

"4. In all stitching operations the presser foot on the sewing machine should have as little pressure as possible on the fabric.

"5. The thread tensions, upper and lower, should both be run as loose as possible. Thread always shrinks upon wetting, and if the tension in the thread is tight, it tends to pucker the fabric, even though it is completely shrunk.

"6. When stitching, the operator should not pull the fabric in an endeavor to make it guide evenly.

"7. When the finished garment is to be pressed, a light spray only should be used. If the garment is to be ironed by hand, the ironer should not stretch the garment during the ironing process. If a machine press is used, the wrinkles in the garment should be smoothed out but the garment should not be stretched.

"8. All new fabrics should be tested for potential wash shrinkage by washing before manufacturing material into garments. *At least one garment should be washed and pressed in a commercial laundry* to positively determine that proper size has been provided and as a final check on the material used."

Avoidance of Misleading Terms in Advertising

The term "preshrunk" or its equivalent as it is directed to consumer should be clearly defined in order to avoid misleading statements in advertising. Already shirt manufacturers, through the National Association of Shirt Manufacturers, have made a positive step in this direction by subscribing to the following recommendations:

1. The shrinkage of a shirt made from preshrunk material when properly laundered shall not exceed 2%.

2. Where the word "preshrunk" or its equivalent is marked on the shirt this shall be interpreted as applying to the entire garment and shall indicate a shrinkage of not more than 2% when properly laundered.

3. When the words "preshrunk collar" or "preshrunk band" or its equivalent is marked on the shirt it shall apply only to the part named and shall indicate a shrinkage tolerance for this part only, of not more than 2% when properly laundered.

Commercial Standards of Sizes

Closely associated with the use of consumer-satisfying preshrunk fabrics is the need for standardization of minimum sizes. Unless minimum sizes become more standardized as time goes on, misunderstandings relative to shrinkage may arise between consumers, retailers, and laundry-owners. Accompanying such standards of minimum garment sizes should be the maximum allowable shrinkage that is permissible in preshrunk cloth that is used for tailoring any specific type of garment. Standards of maximum shrinkage are necessary in order to avoid confusion. Obviously a shirt, for example, should not be sold as "preshrunk" just because the collar has been tailored ¾ of an inch over size to allow for shrinkage. With the establishment of acceptable and workable tolerances, it will be impossible for a garment to be sold as preshrunk merely on the basis of having been tailored greatly over size, a practice that is a make-shift in any event.

The Necessity for Using an Approved Shrinkage Test

In order to avoid confusion, it is absolutely essential that all laboratories making shrinkage determinations use a test that is comparable with that used in any other laboratory. Furthermore, for a laboratory shrinkage test to be a success it should be the equivalent of actual laundering. No laboratory test is satisfactory that produces shrinkage less than that secured during laundering.

The following shringage tests may be used:

1. Three commercial launderings, followed by processing, in the classification in which the fabric would fall in regular laundering procedure.

2. Investigators will find the Standard Method for Determining Shrinkage in Washable Woven Cotton Fabrics, as developed by the American Association of Textile Chemists and Colorists, satisfactory for laboratory tests.

WETTING AGENTS IN TEXTILE PROCESSING

(Continued from page 618)

its adsorption is increased in acid solution by Gardinol because of the strong adsorption of the negative ion of Gardinol. This is entirely consistent with Bancroft's statement that a readily adsorbed anion will increase the adsorption of a basic dye and decrease the adsorption of an acid dye. However, in alkaline solutions the adsorption of the basic dye is decreased by the Gardinol. This is apparently in contradiction to the rule just stated, but is not so in reality since, of course, an anion is not "readily adsorbed" by wool in an alkaline solution. The ability of Gardinol to decrease the adsorption of the basic dye in alkaline solutions may

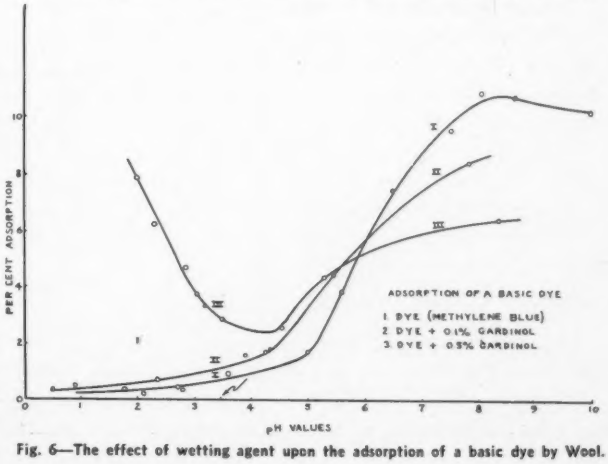

Fig. 6—The effect of wetting agent upon the adsorption of a basic dye by Wool.

be attributed to its peptizing or detergent action, or its negative ion (or micelle) may be thought of as competing with the negatively charged wool for the dye cation.

References

[9]Briggs and Bull, J. Phys. Chem., *26*, 845 (1922).
[10]Bancroft, "Applied Colloid Chemistry," McGraw-Hill Book Co., New York, 1926, pp: 133-136.

SIMPLE METHOD FOR EMULSIFYING OILS

(Continued from page 590)

Also, when the ingredients are in the right proportion the emulsions can be made up with mechanical mixers. Both high and low speed types have been used. The former are liable to swell the mass to a large volume with air bubbles which is usually undesirable.

The following comments are made apropos of certain literature references which will not be cited. In two of the examples given above the soaps were made recently but in the third the oleate mixture was at least four years old. Intermittent stirring may or may not be an advantage but continuous vigorous agitation is not at all necessary. Emulsification on the small scale is readily performed with a mortar and pestle, although the present writer has not tried it.

It is not necessary to put such emulsions through a homogenizer or colloid mill in order to use them for many purposes. In fact several emulsions which were stirred by hand just as described were put through a colloid mill and it made no improvement in them. The oil globules were not made smaller and the milky emulsions were not any more stable to creaming. Neither "broke" with the separation of oil.

It appeared from trials on several kinds of mills that they were of doubtful advantage, at least for the particular materials under investigation. On the other hand a homogenizer did give a finer dispersion of the oil and a greater stability of emulsion.

In making hand mixes, if it is particularly important to avoid the separating of any small amount of free oil from the emulsion, the inside upper part of the beaker in which it is mixed should be carefully wiped before dilution in order to remove any free oil that might appear later and be attributed to "breaking."

Also, ammonium emulsifiers may break down if hot and liberate oil, or free oil may appear on the surface from drying out. Tests for stability should, therefore, be conducted in closed containers.

There may be creaming although no cream line is visible. Simple tests for creaming are to tilt the glass container or dip in the finger. The emulsion will drain off instantly if there is no cream, or the cream will show clearly if present.

Various Materials Used

This work employed mainly the oleates as soaps. Sodium oleate was not very satisfactory; potassium oleate made some excellent emulsions; ammonium oleate is good and may be considered better than potassium. This seems to correspond with the literature. However, for most purposes where there is no particular restriction on the kind of soap that may be used, a mixture of potassium and ammonium oleates is recommended. In the absence of any specific knowledge as to the best proportions they have been mixed in equal amounts on the basis of oleic acid content.

The amounts of soap used are rather high. The lower limits have not been determined because it was not necessary for the work in hand. A moderate amount of soap is usually not objectionable in an emulsion for softening or lubricating textiles.

The reader may judge from a list of soaps and oils that have been used successfully in various experiments, as to the general applicability of the principles outlined:

Ammonium soap from cocoanut oil fatty acids.
Ammonium and potassium oleates.
Triethanolamine and monoethanolamine oleates.
Cocoa butter soap.
Cocoanut oil.
Hydrogenated cocoanut oil.
Japan wax.
Crisco.
Castor oil.
Blown castor oil.
Mineral oil.
Carapa oil.
Olive oil.

An acquaintance who became interested in this procedure applied substantially the same method to the emulsification of a number of waxes by melting them and maintaining the temperature of the mix at about the melting point of the wax in each case.

It is regretted that more positive and general statements cannot be made in some instances, but a little thoughtful work on emulsions will show that there are numerous variations from each case to the next. Good passable results can be obtained without extreme attention to detail, but to make the best possible emulsion in any given case, and to meet any specific requirement such as a definite proportion of soap to oil will require careful study by repeated experiment. The results will undoubtedly vary a little from one sample to another of the same kind of oil.

This scanty treatment of such a broad subject can be considered to be little more than an introduction to it. It would apparently be well worthwhile if somebody could take up the subject of emulsifying representative oils by such elementary methods as have been outlined, and determine more closely the details essential to the best results.

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